

**Process parameter definition for continuous dyeing and
thermofixation based on Dianix[®] disperse dyes on
Polyester fabric with aNIR[®] technology**

Master Thesis

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1.1 Introduction

Polyester which can also be called Polyethylene terephthalate (PET) is a manmade or synthetic fiber originated from air, coal, water, and petroleum. This synthetic fiber has gone through phases of evolution since 1926 from its dyeing, production, manufacturing, and many more. PET goes through different production processes before mostly colorless fibers are formed. Numerous dyeing processes are available in the industry where any can be used depending on many other factors as well.

Continuous dyeing is a two-stage operation which involves continuous preparation and continuous dye application, convection drying and thermofixation which means instantaneous physical application of dye by padding. The dye application padding mangle is the most important component for shade uniformity without giving selvage to selvage or center to selvage shade variation.

In the last decades minimal application technology for continuous dyeing e.g., kiss-roll, ink-jet printing or spraying is used for dye liquor application as well. Besides optimized convection techniques, different radiation sources like UV radiation, infrared radiation, etc. are used for drying and thermofixation.

Application of dyestuff or dyeing polyester fiber is commonly done using disperse dyes because of the hydrophobic nature of polyester. Disperse dyes with its non-ionic nature are volatile, sublime and the dye vapor are intensely absorbed by hydrophobic fibers making it the basics for thermosol dyeing [1]. DyStar Group's Dianix[®] XF2 (high energy) and Dianix[®] CC (medium energy) disperse dyes which introduce properties after dyeing with polyester fiber is the main disperse dyes use in this study.

Drying and thermosoling after dyeing of polyester fabrics with the convection process has already been researched in many studies as to how to make these process steps more significant, energy efficient, sustainable, and many more advanced discoveries.

As said earlier usage of radiation has always been a promising technology to optimize drying and thermofixation in the convection process. Although, radiation sources look promising in such a process, it has always been a question of which combination of types of radiation,

application process and set parameters on a machine can yield the best result compared to the convection process.

In the radiation spectrum, infrared radiation is known to be a solution topic in several areas of research but there still exist gaps for further application. Infrared-radiation is also called heat radiation because it produces the sense of warmth from non-glowing hot bodies [2]. One can use the heat emitted by infrared radiation to dry a substrate in industrial processes. Adphos Digital Printing GmbH discovered a radiation near to infrared which may be used to dry a variety of different substrates. **AdvancedNearInfraRed[®]** (aNIR[®]) radiates from peak wavelengths ranging from 0.78 – 1.5 μm . The aNIR[®] spectrum contains visible light and UV-radiation that are absorbed by water in textile fabrics.

The topic of this thesis is to investigate if this type of radiation is suitable for the continuous disperse dyeing of polyester fibers. Within this work, specific parameters, and temperatures, which are essential for the aNIR[®] radiation dryer, must be identified, so that this technology can be evaluated for its sustainable innovation potential for the industry and our environment.

2.0. Background and state of the art

2.1. Polyester fiber

Polyester fiber is also known as Polyethylene terephthalate (PET). These are fibers that comprise of an ester group which serve as the link between the hydrocarbon or aromatic chain segments of the polymer [3]. The existence of aliphatic and aromatic parts in macromolecular chains and the molecule structure of the fiber makes it distinctive from other synthetic fibers. It is the most accepted thermoplastic fiber within the family of polyesters and has the largest share in production quantity of all textile fibers [3]. This is not simply because of the good end-use properties but exceptionally because it can be easily modified both physically and chemically, reducing negative and enhancing the positive properties.

In 1926, research about synthetic fibers was conducted, which was led by W.H. Carothers whom at that time was working in the United States based DuPont™. The research yielded the discovery of nylon and was continued until in 1946, where DuPont™ purchased all legal rights of the discovery of polyester fiber from Imperial Chemical Industries (ICI) [4]. This led to the production and commercialization of polyester under the name Dacron in the year 1953 [5]. This type of polyester, and numerous options is now produced by different manufacturers thus making poly (ethylene terephthalate) one of the most important fiber polymers [6].

The most important characteristic is polyethylene terephthalate (PET) poly (oxy-1,2-ethanediyloxycarbonyl-1,4, -phenylenecarbonyl). PET has a cost-effective synthesis from petrochemical raw materials (ethylene and toluene) where the fiber can be achieved by high effective melt spinning which makes it display high valuable textile fiber characteristics [3](Figure 1).

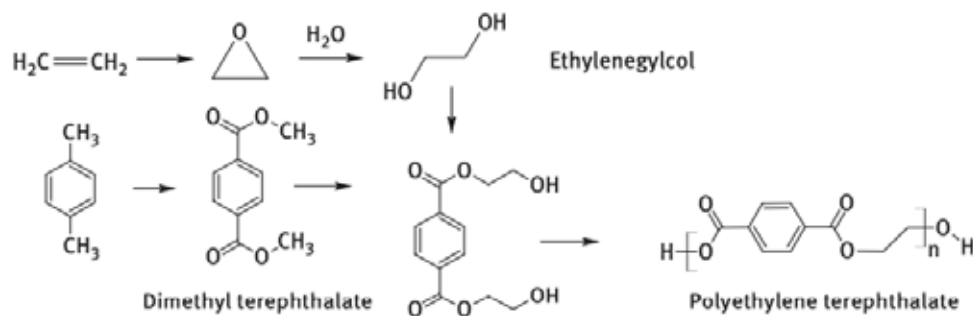


Figure 1: Synthesis of polyethylene terephthalate [3]

To produce PET fiber, a step-growth polycondensation from terephthalic acid (TPA) and ethylene glycol (EG) at higher heat is done involving two starting reaction combinations: esterification and transesterification.

Terephthalic acid (TPA) is the oxidation of methyl group of p-xylene which is repressed with the aid of added bromine-containing promoters as cocatalysts [7]. The oxidation occurs in the air and produces raw terephthalic acid which dissolves at high temperature under pressure in water, hydrated, and purified [7]. Ethylene Glycol (EG) is produced commercially by reacting ethylene oxide with water thus attained by oxidation of ethylene. It is a necessary requirement for smooth polycondensation to poly (ethylene terephthalate) [7].

Esterification reaction is terephthalic acid (TPA) reacted with ethylene glycol at 240 to 260°C with pressure between 300 to 500kPa forming bis(hydroxyethyl) terephthalate (BHET) as prepolymer [8]. Transesterification includes the substitution of methanol in dimethyl terephthalate (DMT) with glycol molecules. This can be done by removing the methanol through distillation where the balance moves at a direction to form bis-glycol terephthalate [3]. Afterwards, ethylene glycol as the byproduct of catalytic polycondensation is instigated at a higher temperature and removed by vacuum distillation. Some catalysts used for polycondensation are antimony (II) oxide (Sb_2O_3) or other oxides of amphoteric materials [3]. The polymer can then be extruded into highly amorphous chips and dried or directly transferred to melt spinning.

Degree of polymerization (DP) refers to the total number of basic structural units including the end group which are present in the polymer [9] [10]. The DP in PET has a measurement of 80-150 formula units. Polycondensation reaction follows statistical rules, thus the polymer mass has a significant amount of low molecular-weight oligomers present. Corresponding values are as much as 2wt% of cycle trimers and linear oligomers [3]. Throughout a textile process like high temperature dyeing, low-molecular weight substances are discharged into the process bath. This can cause uneven dyeing, an effect on the appearance of the dyed material and technical problems at the sites of the low temperature (valves), if the deposition of PES oligomers to these areas is not attended [3].

Molecule interactions amongst the chains are established on dipole-dipole interactions between carbonyl groups of ester bond and on π -bond interactions between aromatic rings [3].

The aromatic rings are positioned parallel in the matrix of PET allowing strong π -electron interaction. This clarifies the short chain length of the polymer which sufficiently produces high fiber strength.

Comparing the degree of polymerization (DP) with other polymers such as cellulose, should be considered that one formula unit in ethylene-terephthalate is more than twice as long as an anhydroglucose unit, therefore these two polymers are not to be compared only with the DP values [3].

PET molecular end groups can either be hydroxyl or carboxylic groups. The average value for the carboxylic group content of cellulose is comparable to PET which has a measurement of 10-40 mmol/kg of polymer. The existence of acidic groups explains the possibility of acid-catalyzed ester hydrolysis at higher temperature of around 270°C which are applied during fiber extrusion. Therefore, humidity content of raw PET polymer must be less than 0.01wt% to prevent the reduction in DP [3] (Figure 2).

Spinning of PET fiber is normally achieved under higher temperatures above the melting point, that is in the range of 225 and 265°C. This range is dependent on crystallinity and the crystal size of the polymer. The fibers are extruded through a spinneret and drawn in length of a factor of about 2. Properties of the fiber are subjected to change during this some process. Especially when it has an increase in the longitudinal orientation of the fiber, increase in crystallinity, and increase in tenacity.

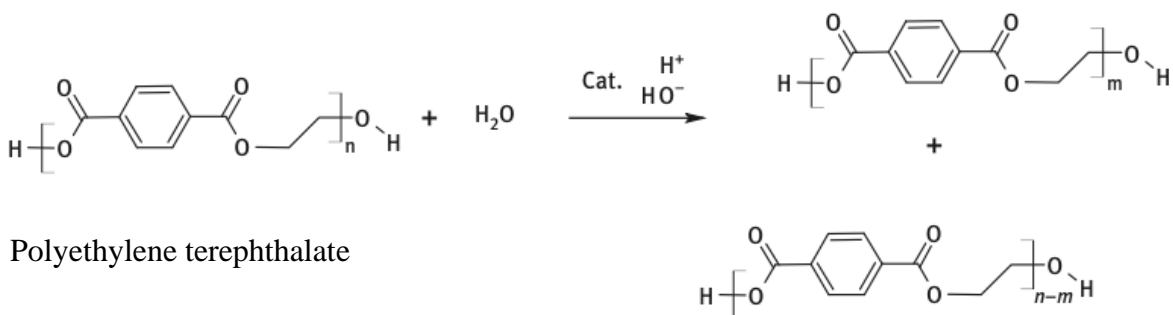


Figure 2: Hydrolysis of polyethylene terephthalate [3]

During melt spinning, a drawdown tensioning is done to create a molecular orientation in the filament; the orientation is frozen during cooling where amorphous and other flexible parts of the polymer chain stay weak. Because of PET's high glass transition temperature (70°C), the spun material solidifies completely in the spinning shaft to form a stable, cooled melt with molecular orientation in the yarn direction [11].

The orientation is relative to the tensile stress alongside the path of the yarn during the time and moment of solidification [12] [13] and this makes PET demonstrate a distinct characteristic which separates it from other commercial melt-spinnable polymers [7].

At the time of solidification, the filaments reach their final winding speed and attain their final as-spun fineness. The point of solidification, which rests alongside the as-spun yarn, is dependent on spinning conditions between 20 and 150 cm from the spinneret [13]. The as-spun yarn is amorphous if the drawdown tension is not better, and this is bound to happen due to the relatively low tendency of PET to crystallize at room temperature. Stressed-induced crystallization happens exclusively at higher tensile stress because of spinning speeds above ca. 3000 – 4000 m/min or of very intense air quenching, where the crystallinity is also frozen into the yarn. Then, the as-spun yarn is partially crystalline under these processing conditions [14] [15].

At extremely high speeds of spinning around 8000 m/min, crystallinity of the as-spun yarn becomes finer and can be as high as 50 % [16]. At spinning speeds greater than 4000 m/min, there is sheath-core placement added in the filament [16].

This effect can cause the chain orientation in the sheath, measured by birefringence, to be nearly two times greater than in the filament core [17]. Consequently, the sheath of the filament is highly crystalline, and these differences continue in the further processing of the as-spun yarn and can lead to non-uniformities in the dyeing process, if they are not controlled [18].

The effect of spinning speed dominates if spinning speed is increased and the fineness is adjusted by relatively increasing mass transport. This can cause the crystallinity and the orientation to increase [13] [15]. When the spinning speeds are less than 5000 m/min, the diameter of the spinneret holes, meaning the spin-to-draw ratio, has less effect on the final spin orientation and crystallinity [19].

PET can be produced either continuously meaning the spinning and polymerization components can be linked together or produced batchwise. Direct spinning has economic advantages producing high-volume end products, e.g., for staple fibers and standard filament yarns. [20].

For batchwise production, firstly, PET granulate is conveyed to a spinning plant and stored in silos. The granulate needs to be dried immediately before melting to avoid hydrolytic degradation of the melt because of the minutest amount of water present [7]. The granulate is dried, while it is moving slowly downward, by a hot countercurrent airstream blowing over it in continuous shaft dryers. A crystallizer in which the chips are slightly crystalized on the surface normally heads the dryer to prevent the amorphous PET to draw out in a sticky state [21].

There are several dryer designs which have throughputs that amount to numerous tons per hour. Drying the PET granulate in batches in a large tumble dryer or in a vacuum is better for some products [7]. The granulate gets to the melt extruder after the dryer and is then transferred to one or more spinning machines. Most importantly, maintaining equal hold-up time in the hot line between the extruder and attached spinning units need to be achieved to avoid thermal degradation of the melt. The temperature of the hot lines must be the same to make the as-spun yarn and the end-product uniform. There are different spinning units in construction and processing data depending on the end-product. The same is said for the method of cooling, drawing down, finishing, and collecting the as-spun yarn [11]. Industrial yarns have a common operational winding speed of about ca.500 m/min and 6500 m/min for filament yarns which are spun at high speed with both having a temperature of around 270 – 310°C. Monofilaments are normally spun at lesser winding speeds [7].

Drawing of an as-spun PET yarn is always done at a temperature greater or equal to 80°C commercially and this is because of additional process of drawing. The yarn temperature in the drawing zone is relatively higher than exterior temperature which is sometimes dependent on the arrangement of the drawing [7].

Throughout drawing, the molecular network of the as-spun yarn is oriented further, and the amount of parallel molecular chains is increased which starts the crystallization of the PET fiber. The more the fiber is drawn, the more oriented the crystalline and amorphous regions become.

Crystallinity and strength increase with increasing draw ratio while break elongation decreases in the drawn yarn [22]. This is illustrated with a stress strain diagram (Figure 3) where two yarns are drawn differently.

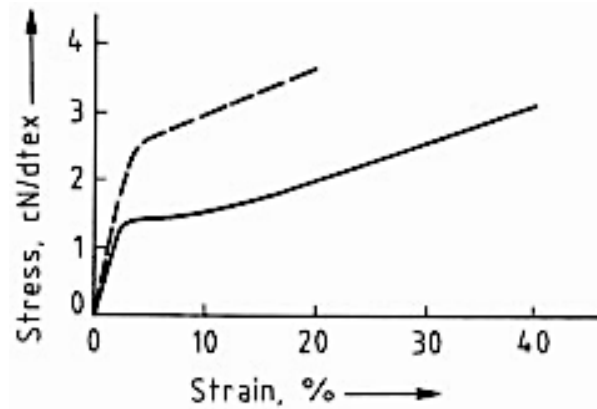


Figure 3: Stress-strain curves of two drawn PET yarn
Drawn ratio 1:1.52; Drawn ratio 1:1.83 [22]

PET can be drawn more and more easily depending on how much the drawing temperature exceeds the glass transition temperature [12]. Industrially, PET as-spun yarn is always drawn continuously between two set of rolls which has its speed controlling the draw ratio [23]. By this practice the required temperature is conveyed to the yarn by heated rolls [11].

Thermal instability of drawn PET yarn structures are quite difficult to work with during further processes or textile end products, therefore the thermal instability is reduced during the fiber and yarn production by a thermal aftertreatment, called heat-setting [11] [24]. The temperature at which some types of heat-settings are carried out is frequently very high, about 220 °C. Cooling the material under tension, especially when it leaves the heat-setting zone, is very important to prevent shrinkage.

Heat-setting can be done before, during, or after dyeing. Heat setting done before dyeing will increase the dye uptake in most of the cases. Heat setting of polyester influences the crystallinity of polyester fibers. Also, water, steam and carrier influence the amorphous structure of polyester fibers as well. Both influence the dyeing behavior of the fabric. Other targets of heat-setting are highest dimension stability, reduction of tensions, removal of fabric related wrinkles which gives a constant and defined fabric weight.

2.2. Disperse dyes

The Society of Dyes and Colorists (UK) defines disperse dyes as a substantial water-insoluble dye having substantivity for one or more hydrophobic fibers, e.g., polyester, or cellulose acetate and is usually applied from fine aqueous dispersion [25].

Dyes before the world war I were applied from solution in an aqueous dyebath to substrates such as wool, cotton, silk, and other natural fibers [26]. After man-made fibers were introduced like cellulose acetate, with its inherent hydrophobic nature, it created a setback where few dyes could have affinity to these new manmade fibers [1]. Research conducted by Green and Saunders developed the Ionamine Dyes (British Dyestuffs Corporation) in 1922 [1](Figure 4). These water-soluble dyes were hydrolyzed in an aqueous dyebath to create a carefully fine suspension free base for the fiber to absorb.

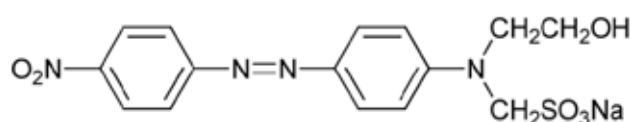


Figure 4: An Ionamine dye [1]

In 1923, Holland Ellis developed Sulphur Resolonic Acid (S.R.A) colors or dyes where S.R.A was a dispersing agent. This dispersing agent along with the dyes put together in an aqueous solution resulted in a very fine dispersion of the dyes [27]. British Celanese Corporation and British Dyestuffs Corporation examined independently how the aqueous dispersion of dyes could improve which yielded results of different ranges of disperse dyes like Duranol[®] Imperial Chemical Industries (ICI) and SRA superseding the earlier dyes [1](Figure 5).

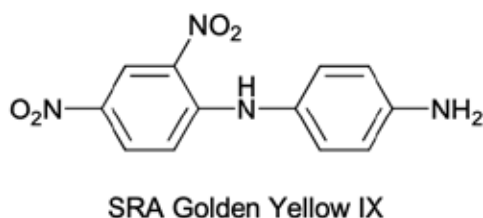


Figure 5: SRA range dyes [1]

Disperse dyes with its non-ionic nature are volatile and the dye vapor is intensely absorbed by hydrophobic fibers, which is the basis for thermosol dyeing and heat-transfer printing process [1]. These dyes have more than 50% made of simple azo compounds, about 25% anthraquinones, and the rest are, nitro, methine and naphthoquinone dyes [28]. Disperse dyes were originally made for cellulose acetate fibers.

During dyeing at 85-95°C, the required dyes must be small molecular sizes, which have a satisfactory solubility and diffusion into the fiber [1].

These dyes possess high vapor pressure, that cause no drawbacks. During the introduction of polyester fibers, problems of fastness developed due to the dye sublimation during the subsequent heat setting and pleating processes. The relative molecular mass of disperse dye is related to the vapor pressure of the dyes and can be decreased by increasing the molecular mass of the dye and by including polar substituent groups in the dye molecule, such as amide groups [1]. Including of polar groups in the dye molecule produces a minor increase in water solubility in addition to increasing the molecular mass which results in slower rate of diffusion. Some characteristics of disperse dyes are their crystallinity, with high melting points of more than 150°C [1]. They are mixed with dispersing agents to get an even dispersion in a dyebath. They are to disperse quickly when added to water and must have very fine and uniform particle size. The dispersion should be stable even with the addition of auxiliaries like carriers and leveling agents.

Disperse dyes come in the form of powder or liquid aqueous dispersion. In the powder form it is comprised of dispersing agents such as naphthalene sulphonic acid formaldehyde condensate and maybe wetting agent [1]. The liquid form has the advantage of freedom from dusting, easy to prepare dyebath and padding liquor and also the suitability for automatic metering systems named 'auto-dispensing' [1]. They are comprised of less amounts of dispersing agent and produce higher color yield and brighter dyeing in pad-thermosol process. The saturation level of pure disperse dyes in the fabric are relatively high and due to the absence of ionizable groups, they have the trend of subliming without decomposition. Sometimes, fading of the color of these dyes may come from ironing [1]. Disperse dyes exhibits thermal migration where dyes migrate from the core of the fiber to the surface during drying of the goods at 170°C or above [1]. Low wash fastness is a drawback in thermal migration which can be solved by post-scouring the dyed fabric and drying at 140°C or below.

2.2.1. Classification of Disperse dyes

Imperial Chemical Industries (ICI) created a methodology for the classification of disperse dyes in the 1970's according to their dyeing properties and sublimation fastness, enlisting them into categories A to D [1]. Class A dyes had relatively low molecular masses, which exhibit poor sublimation fastness but desirable color fastness [1]. Class D are relatively high molecular mass, exhibiting good sublimation fastness but poor color fastness. BASF also pronounced their own comparable system of disperse dye classification. The Disperse Dye Committee of the Society of Dyers and Colorists classified the dyeing characteristics of disperse dyes according to results of the test which can be performed on the dyes [1]. Tests were performed on dyes to evaluate their characteristics like leveling properties, build up, rate of dyeing and temperature range of the dyes on hydrophobic fibers.

There are two types of disperse dyes which are high energy disperse dyes and low energy disperse dyes. High energy disperse dyes are more polar, high molecular weight dyes and have poor migration during dyeing but good heat and sublimation fastness, e.g., DyStars' Dianix[®] XF2 disperse dyes. While low energy disperse dyes have lower molecular weights, with low polarity, are rapid dyeing dyes with poor sublimation fastness [29].

Dye chemists who aim to produce dyestuff, find the chemical classification by chromogen very valuable. In 1992, Color Index registered around 1,150 disperse dyes by their chemical classes or structure which are azo, anthraquinone, methine, nitrodiphenylamine, aminoketone, quinoline and miscellaneous.

Azo dyes are compounds which are comprised of at least one azo group attached to sp^2 -hybridized carbon atoms like naphthalene, thiazole, benzene and thiophene. Azo dyes are present in the more stable planar trans-form thus making the carbon-nitrogen bond angle approximately 120° [30](Figure 6).

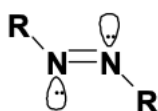


Figure 6: The trans-form of azo dye [30]

Azo dyes are the most important class, comprising more than 50% of the total commercialized disperse dyes worldwide. These classes of dyes dominate because of their intense chromophore, regarding the 2 or 3 times stronger tinctorial strength compared to conventional weak anthraquinone chromogens and their susceptibility to reduction clearing (for details see Chapter 2.6.1.). They cover the yellow, orange, and red hues. The important subgroup is aminoazobenzene derivatives (Figure 7) [31] which include

R_1 = electrophilic group

R_2 and R_3 = H or electrophilic groups

R_4 and R_5 = H or nucleophilic groups

R_6 and R_7 = H or alkyl or substituted alkyl groups

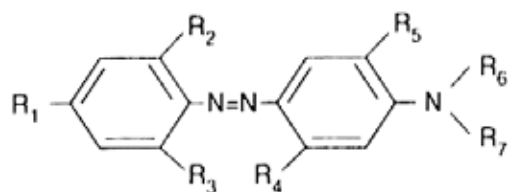


Figure 7: Aminoazobenzene dye [25].

Anthraquinone disperse dyes are a large group which cover the greenish yellow to blueish green hues [25]. Simple anthraquinone dyes are bright and stable under dyeing conditions. These structure of disperse dyes are derived from anthraquinone-a-sulphonic acid which requires a mercury catalyst. This creates pollution problems which made alternative methods of manufacturing available [25], some examples are C.I. Disperse Violet 1 (Figure 8), C.I. Disperse Blue 56 (Figure 9) and more. They are weaker in tinctorial value compared to azo dyes.

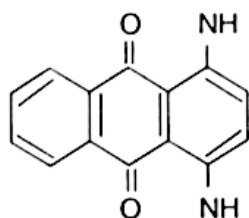


Figure 8: C.I. Disperse Violet 1 [25]

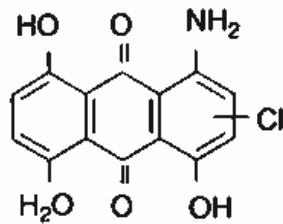


Figure 9: C.I. Disperse Blue 56 [25]

2.2.2. Dyeing mechanism of disperse dyeing

Dyeing of hydrophobic fibers, like polyester, with disperse dyes may be considered as a dye transfer process from liquid solvent (water) to solid organic solvent (fiber) [32]. These dyes are added to water with surface active agent to form an aqueous dispersion. There is a change in the state of a low molecular weight crystalline solid when heated up to its melting point. The dye molecules then move into the amorphous regions of the fiber where they are held by hydrogen bonds and Van Der Waals' force [32].

Polymer molecules are not easily separated due to their length preventing the free separation of the molecules but rather segments of polymer molecule can be separated as the temperature is raised. The temperature at which chain segments start to separate is termed the 'glass transition temperature' or T_g . [25]. When this temperature is surpassed, the frequency of the change in the position of polymer segments, also known as segment mobility, rises with the increasing temperature. This leads to a fast increase in the rate of dyeing [25].

Firstly, dispersing of the dyes from its solid state in the water into individual molecules. This diffusion of the dye in the dye bath depends on dispersibility, and solubility of dyestuff. The dispersion is stirred using dispersing agents and higher temperature. (Figure 10 Sec.1,2).

Adsorption of the dyes from the aqueous solution onto the fiber surface happens and this is subject to the solubility of the dye in the dye bath and their affinity to the fiber surface (Figure 10 Sec.3).

The dyestuff adsorbed on the surface of the fiber now diffuses into the interior towards the center of the fiber by raising the temperature (Figure 10 Sec.4). The dyes molecules are then fixed by forming bonds between both. In case the equilibria stage following section 1 to 4 is not interrupted by follow up reactions, uneven distribution of dye molecules can be levelled out in this stage of dyeing (Figure 10 section 5).

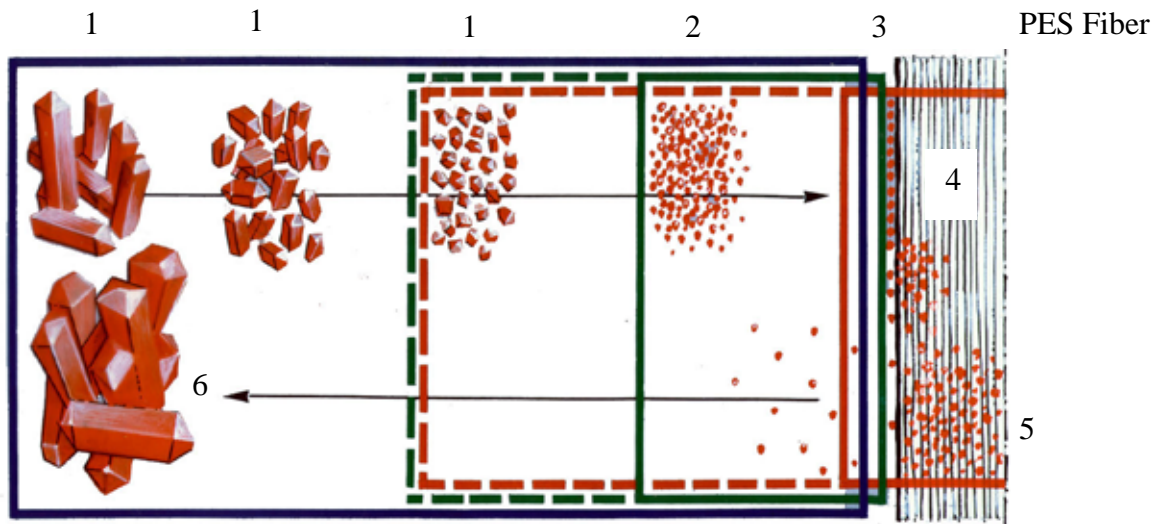


Figure 10: Dyeing kinetic of disperse dyes. Source: DyStar® Group

Section 1	Dyes in dispersion
Section 2	Dyes in monomolecular dissolved phase
Section 3	Dyes adsorbed to the fiber surface
Section 4	Diffusion of dye particles
Section 5	State of equilibrium of dyes
Section 6	Recrystallisation of dyes

Disperse dyes give uneven dyeing when used directly in dye bath but this can be solved by the introduction of special chemicals named dispersing agents. These dispersing agents must be effective under dyeing conditions, i.e., high temperature and hard water [32]. Dispersing agents contribute to the dye particle size reduction and maintain the dispersion in the dye bath during the dyeing process.

The temperature plays an important role during the dyeing process. For high temperature dyeing methods, 100°C temperature is required for the softening of the fiber. In carrier dyeing, temperatures at 80-90°C are required for the swelling and softening of the fibers. An increase in temperature also increases solubility of the disperse dyes even though disperse dyes are almost water insoluble.

2.2.3. Fastness properties

Over the years, more detailed information has been discovered about fastness properties of various groups of closely related dyes which have minute structural changes. The relationship between the chemical structure of a dye and its fastness properties has been complex and difficult to trace. An acceptable color fastness to washing, rubbing, sublimation (fastness to dry heat), light etc. is very important for all dyes [33].

2.2.4. Fastness to washing (color fastness)

Environmentally friendly disperse dyes with high wash fastness on polyester are increasingly on demand in the industry. Fastness to washing depends on the diffusion of the dyes inside or outside the polyester fabric at limited time of washing cycles or tests. Therefore, the fastness will be exposed to kinetic control where the increase in size and polarity of the dye molecules will reduce its mobility within the fiber and then reflected in lower diffusion coefficient and an increase in wash fastness [1]. Also, Fastness depends on the glass transition temperature (T_g) because the dyestuff diffuses only in the amorphous structures.

Polyester fibers with its more crystalline and hydrophobic nature makes it have better fastness to wet treatments like washing and perspiration fastness than acetate and nylon fibers with a certain dye [1]. For a more acceptable wash fastness on polyester, the selection of the dyes is now more important than ever because of the more demanding wash fastness tests used presently and a variety of aftertreatments employed. Before the 1970s, the universal wash-fastness trial for dyed polyester was ISO 105 C03 wash test. This test replicated domestic washing with soap and examined staining on polyester and cotton or wool.

Almost all disperse dyes gave good results but beyond the 1970s, attaining acceptable fastness to wash became more difficult because of customer-driven rise in standards being implemented in the guise of more severe testing protocols [34]. Stringent conditions were stipulated in the 1960s by Marks and Spencer, who desired all their suppliers to use only those dyes that met criteria using adjacent nylon, which showed a greater tendency to stain than wool or cotton. Low wash fastness was also observed when heat treatments or high temperatures implemented lead to thermomigration of the dye molecules. Dyes move from the core of the fiber towards the surface, thus counteracting the reduction clearing [1].

An official range of European wash test (ISO 105 C06 series) was published in 1980s which reproduced the widespread adoption of detergents for domestic laundering with dyed polyester fabrics. This gave lower ratings than soap-based tests. Any dye stripped from the fiber by the soap tends to remain solubilized through the surfactant properties of the soap present in a relatively large concentration [1].

Lately, there are improved wash fastness dye structures in modified dyes containing diesters, thiophenes, benzodifuranones, thiophenes, phthalimides and fluorosulfonyl groups. Dyes containing diester for example, have improvements from wash fastness coming from higher and easier cleaning of the fiber surface. This is not only in after treatment but is also present in saponification. Some of these designed improve wash fastness groups can be seen in (Figure 11) [35] [36].

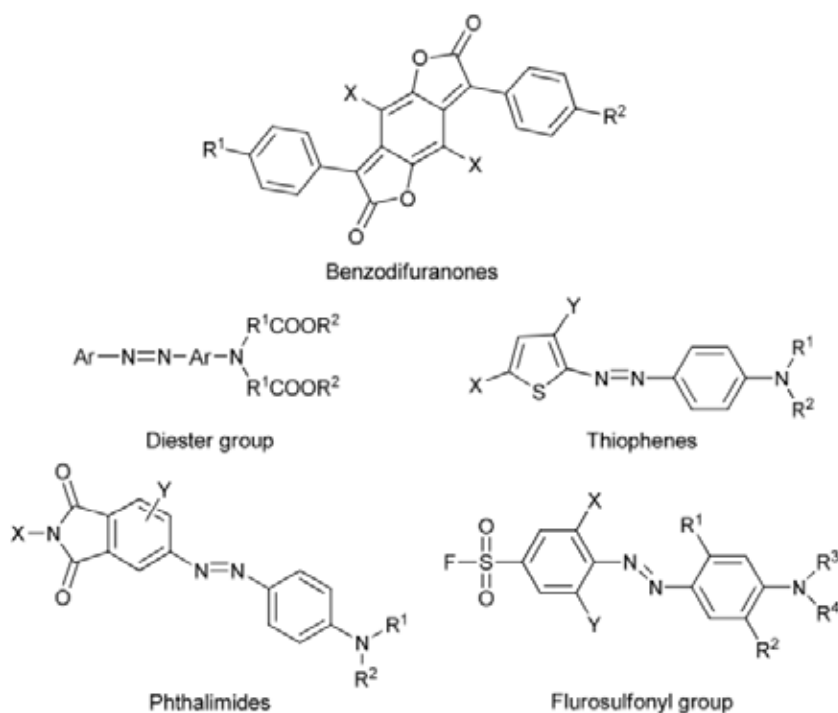


Figure 11: Developed disperse dyes with high wash fastness [35] [36]

2.2.5. Color fastness to dry heat (excluding pressing)

Due to the heat treatment during finishing of polyester fabric after disperse dyeing, fastness to sublimation or dry heat is an essential property that needs to be tested. Disperse dyes, ideally, are small, non-ionic molecules having low molecular weight and as such exhibiting significant vapor pressure. When heat treatments such as ironing, are exerted on disperse dyed fabrics, the dye may sublime and cause contamination on adjacent undyed materials [37]. Increasing the size and/or polarity will reduce the rate of diffusion moving out of the fabric and vapor pressure, which then increases the sublimation fastness [1].

With the same change, the application by exhaustion methods becomes problematic as the low diffusion coefficient gives slow fiber penetration, less color yields and reduced leveling power [1]. Hence, a balance between polarity and reduction in hydrophobicity must be found. Due to the property, Class A and Class D classifications of disperse dyes were made. Class A disperse dyes having excellent dyeing properties but poor heat fastness and Class D having excellent heat fastness but poor dyeing properties [38]. Better sublimation fastness can be attained by increasing molecular size and by also adding hydrophilic groups into anthraquinone rings [1].

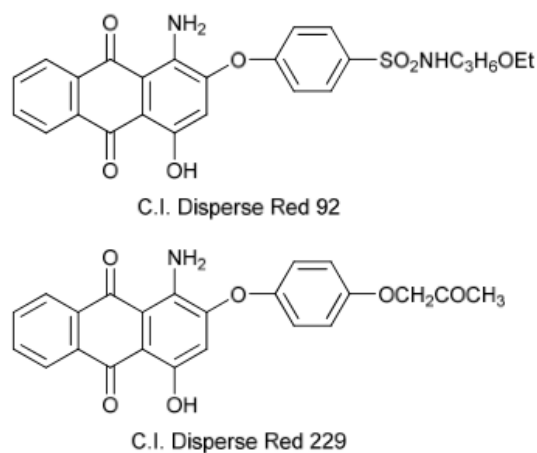


Figure 12: modified dyes for better sublimation fastness [1]

2.3. Polyester dyeing process

In practical terms, only disperse dyes are appropriate to dye polyester fibers because of their hydrophobic properties [1]. These dyes used on polyester fiber provide a wide range of hues with good build-up and fastness properties satisfactory for many purposes. The form in which disperse dyes exists in the dye liquor is a major influence in a dyeing process. Also the water solubility and rate of dissolution are influenced by factors such as temperature, particle size, melting behavior, crystal growth, crystal modification, presence of salts, behavior in dye mixture and type of dispersion and dispersion agent used [39] [40].

The order of dyeing polyester fiber is to dye, rinse, reduction clear to remove dye deposited on the surface of the fiber and the soaping [25]. The rate of dyeing may be raised so that it can be accepted commercially by raising the working temperature to a region of 130°C, or by dyeing at a boil with the existence of an accelerating agent ('carrier') [41]. Transferring of disperse dyes to polyester fiber can also be done under dry conditions by impregnating the goods with an appropriate dispersion, drying, and proceeding with a baking temperature in the range of 190 – 220°C [42].

2.3.1. High temperature dyeing

In batch coloration, high temperature method is the most well-known. The temperature for this method (ca. 130°C) requires increased diffusion of the dyestuff, leading to an increased rate of dyeing. [1]. There three phases of exhaust dyeing are adsorption phase, high temperature or diffusion phase, and clearing phase. (Figure 13)

The adsorption phase is the most important stage where the levelness of the dyed fabric is determined and important that the heating rate is suitable to allow controlled adsorption of the dye molecules. This phase can be influenced by temperature gradient, concentration of dye, fiber type and auxiliaries.

High temperature or diffusion stage is the stage where there is the transfer of the dye liquor adsorption and the molecule diffusion into the amorphous regions of the fiber [43]. In this phase, the general time is 10-20 minutes for dyeing pale shades, 20-30 minutes for medium shades and 30-35 minutes for dark shades. The time required at high temperature is also directly related to the diffusion characteristics of specific dyes and their dyeing depth.

An important property of the dyes used in this high temperature phase is their tendency to level out or dyestuff migration. This is important especially when the dyes have been ununiformly adsorbed in the adsorption phase because of inadequate liquor circulation or a rapid heating rate, then in the high temperature phase the diffusion rate of the disperse dyes becomes a key parameter.

During the clearing phase, the treatment carried out is termed reduction clearing where dyed fabric is treated in a strong reduction solution, usually comprising of sodium dithionite and caustic soda. The satisfactory time and temperature for reduction clearing is 20 minutes at about 70-80°C [1].

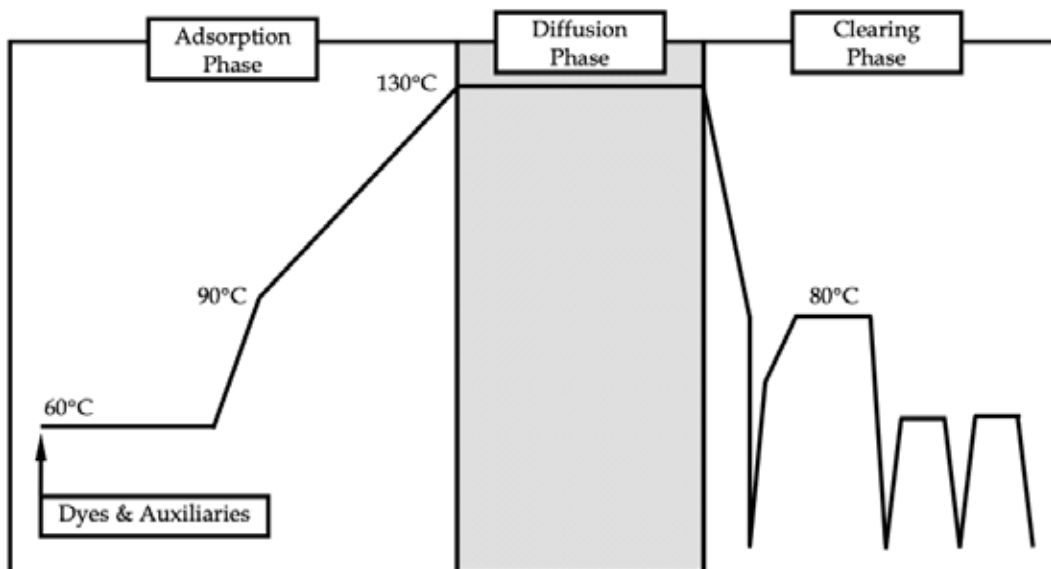


Figure 13: Phases of exhaust dyeing of polyester [1]

2.3.2. Continuous/Thermosol dyeing

The important process for continuous dyeing of polyester and polyester/cellulose fiber blend using disperse dyes is the thermosol dyeing process. This process is used for woven and knitted materials [2]. During the dyeing of polyester fibers, the dyeing time can be reduced drastically by increasing dyeing temperature [28]. The rate of dyeing increases significantly above the glass transition temperature, with a temperature increase of 10°C approximately doubling the rate.

Due to this reason, ca. 200°C fast colored fibers can be achieved with disperse dyes in seconds and especially for polyester/cellulosic blends, the process is used to dye these fibers continuously in a pad-fix process which was introduced by DuPont as an example of the thermosol process [44].

After dyeing or application of dyes on polyester, drying and fixing is next. During drying, dye liquor padded on the fiber surface can migrate into positions with high rate of drying. This hazard can be stabilized using antimigration agents. Drying can be done using hot air dryer or by infrared radiation. After drying the dye is present on the surface of the fiber and is located in a film of dyeing auxiliaries and dispersing agents.

Most disperse dyes are dissolved in the liquid film of auxiliary agents at fixing temperatures of approx. 210°C. Mostly at a temperature of 210°C, the disperse dyes begin to sublime and the polyester fiber adsorbs their vapors [45]. During this fixation, there are three processes which overlap, heating, dissolving of the dye in the fiber surface layer and diffusion into the fiber core.

Dyes of lower molar mass tend to sublime more freely but have a disadvantage of low fastness and poor resistance to heat treatments. Dyes of high molar mass have good fastness properties compared to dyes of low molar mass but are more difficult to apply.

After thermofixation, reduction clearing treatment is essential to remove unwanted dyes remaining on the surface of the fiber.

2.4. Application technology

This chapter features textile machinery and processes. Textile machinery and processes can be classified into different characteristics e.g., dry processing and wet processing [3]. The dry processes include mechanical techniques like sanding, shearing, and brushing and chemical/physicochemical processes like plasma process, laser treatment of denim singeing, etc. [3]. The wet processing techniques include the usage of solvent-based system for textile chemical processes. Some examples are water, printing paste, supercritical CO₂, ionic liquids, foam, steam, and solvent systems like hydrocarbons [3]. It is important for one to know the structure of textile dyehouse and finishing plant according to the classification of machinery regarding applications of processes [3] (Figure 14).

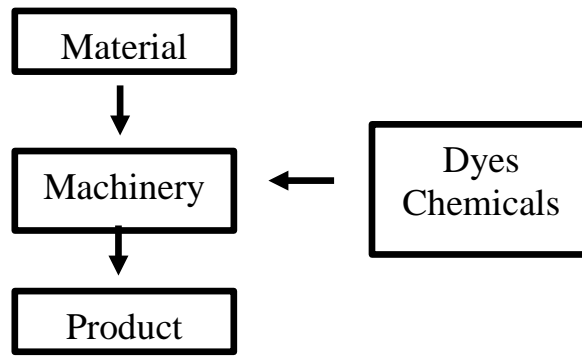


Figure 14: Structure of dyehouse and finishing units [3]

Machinery for wet processing of textiles is intended to be used on some group of textile materials such as fibers, fabrics, and yarns with some few exemptions [3]. These can be done either in a continuous process or discontinuous (batchwise) process. The machine design is made in a way that different process can be produced, but for textile dyeing and finishing industries it is best to specialize on certain group of products to be processed on an available equipment [3]. Normally, the recipes for dyes and chemicals are tailored to the machine available so that the best quality results can be obtained. Some few selected machinery, applications and processes for textile chemical process are discussed in the further chapters.

2.4.1. Padding and Spray application

Padding

Padding or pad process is so far one of the most important application techniques. Padding or pad process involves a dry fabric being passed through a dipping trough of dye liquor solution. The fabric then picks up the dye liquor and then the excess is removed by squeezing between rubber rolls (pad rollers) (Figure 16).

The compression of the pad rollers improves the penetration of the dye liquor into the fabric. This gives a controlled uptake of liquor on the fabric. The resulting liquor uptake, which is the liquor pickup, varies depending on the fabric material from about 40-100 % [46].

Definition of pad liquor pick up can also be explained with this formula

$$\text{Pickup} = \frac{\text{weight of wet fabric} - \text{weight of dry fabric}}{\text{weight of dry fabric}} \times 100 \%$$

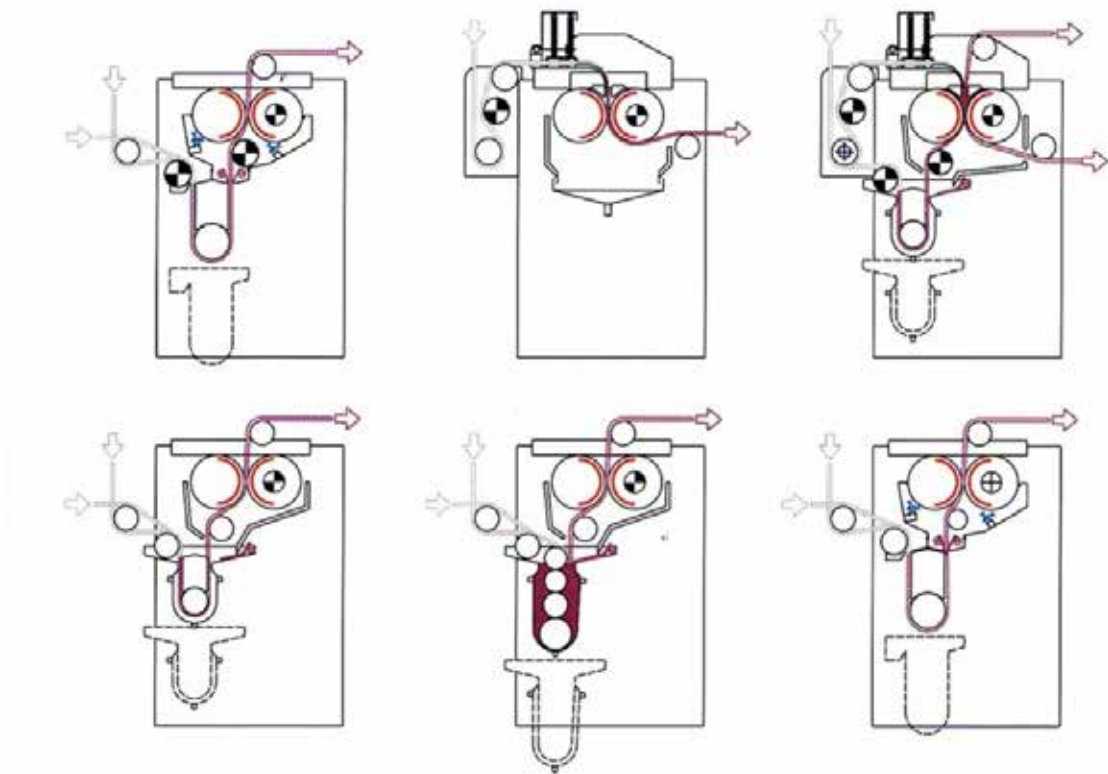


Figure 16: Variation of padding technologies (source: Küsters Textile GmbH)



Figure 15: Lab padder (source: own picture)

The amount of liquor picked up strongly depends on the squeeze pressure produced by the two rollers. The curve of liquor pickup plotted against squeeze pressure is nonlinear (Figure 17) [46].

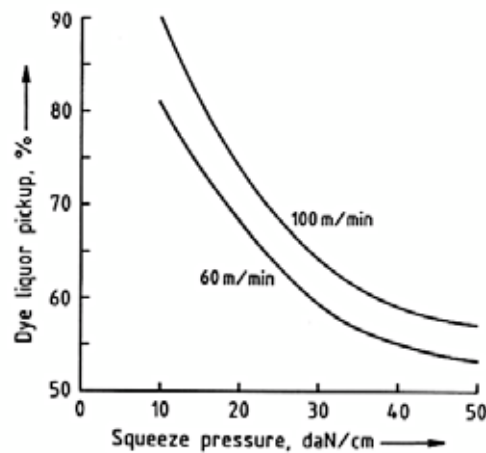


Figure 17: Effect of squeeze pressure of pad rollers on dye liquor pickup at different running speeds (60 and 100 m/min) [46]

A lower liquor pickup is required to reduce the migration effect and cost involved at the drying stage. The pad dyeing process includes the nip of the mangle which makes sure the liquor flows into the fabric with high energy. This results in the fabric having good penetration and less two-sidedness [46]. One of the problems encountered is when the two rolls are squeezed together by means of the end bearings which do not provide a uniform pressure over the full width of the fabric. The reason being bending of the rollers takes place. To resolve this problem is by fitting a bend tube in the body of the tubular roll [2].

Drying dyed fabric is a highly energy consuming process where evaporating 1 kg of water is estimated to require 1 kWh of energy [46]. In natural fibers dyeing process, high water uptake needs more energy than that of synthetic fibers. Due to this, there are techniques which are called 'low-add on techniques' which allow the application of chemicals from more concentrated solutions and reduce energy consumption during drying. These techniques normally prevent the dipping of the fabric in the chemical solution to arrive at a pick-up of around 30 % [46]. Some of these techniques are spraying, kiss-roll, coating, etc.

Spraying

This application method is done with devices (RotaDyer Lab machine at WEKO/RotaSpray) for low wet pick-up with solutions sprayed onto the fabric using nozzle rotating atomizers (Figure 18). The nozzles are controlled by a computer program, programmed for applying the dye liquor on the substrate, and other functions as well.

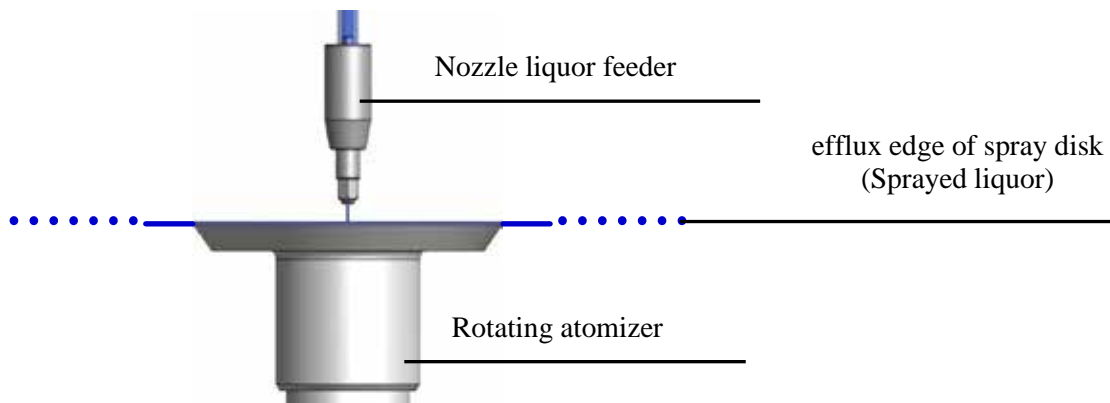


Figure 18: Nozzle and rotating atomizers (RotaSpray GmbH)



Figure 19: RotaDyer Lab machine (source: WEKO/RotaSpray)

After the dye liquor is fed in the system and all control settings are done, the liquor passes through the nozzles and is sprayed. The liquid filament pinches off in the close-up range of the efflux edge which creates micro droplets. The target is to arrive at an equal (monodisperse) drop size distribution of the dye liquor. In principle of the dyeing process, it is a 2 spray contacts back and 2 spray contacts face (Figures 20,21).

- 1- The Rotating Atomizer first gets in contact with the back of the fabric via spraying
- 2- Runs on the rollers above, turning the face to have contact with the Rotating Atomizer
- 3- Runs again on the rollers below and presents the back of the fabric to be in contact with the other set of spray Rotating Atomizers
- 4- Finally, runs on the rollers above and presents the face of the fabric the second time to the Rotating Atomizer

The RotoCoater/RotoDyer has some advantages of

- having Pick-up 10 – 25 % for chemical add-on
- Low energy consumption for drying/curing/crosslinking
- Low carbon footprint
- Higher productivity and reduced total costs

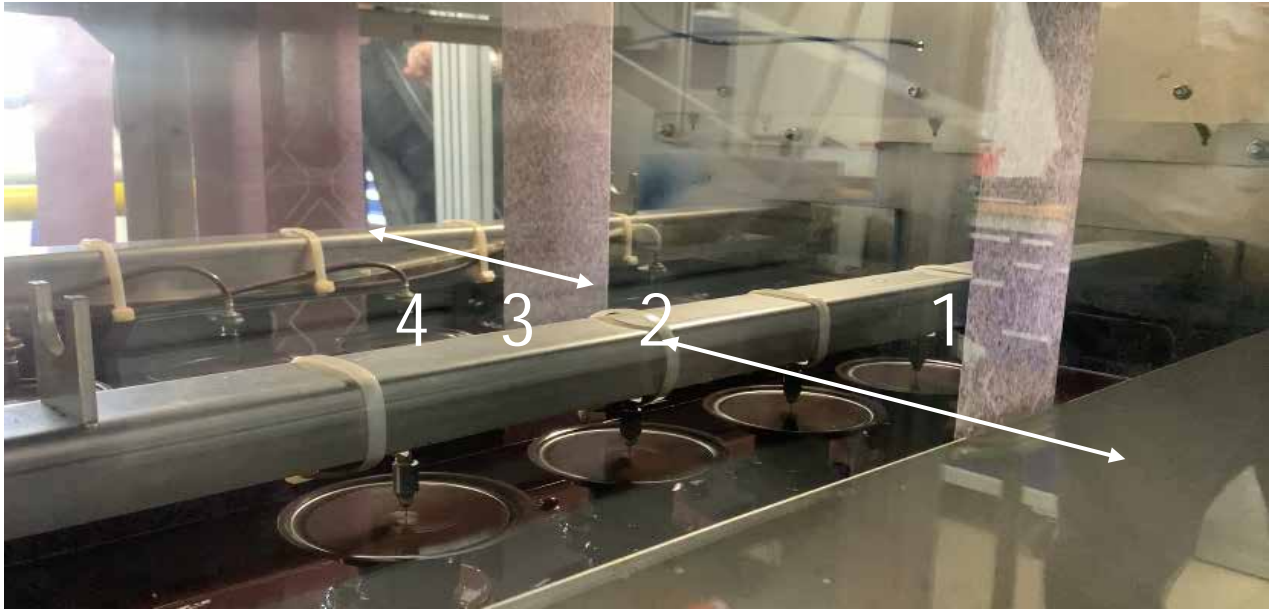


Figure 21: spray principle of RotaDyer Lab machine (own photo)

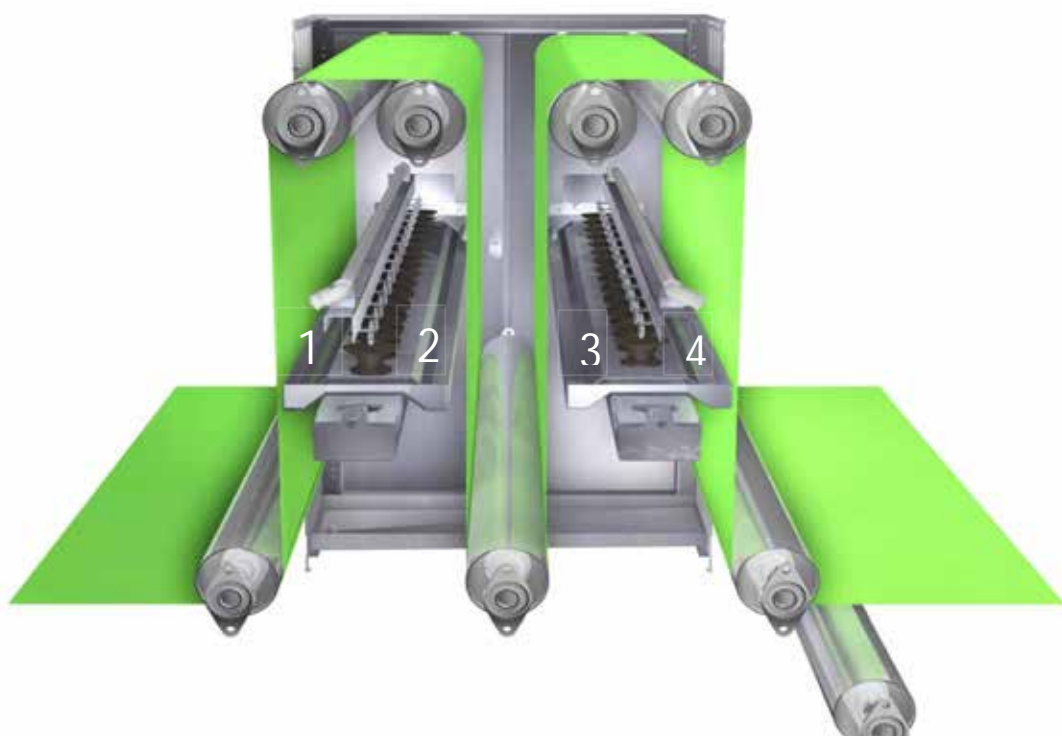


Figure 20: Schematic image of the spray principle RotaDyer Lab machine [47]

2.5. Conventional drying and fixation process

Drying is the stage between wet processing and dry finishing [2]. Drying of wet textile fabric can be done with different machinery. Drying can be achieved in heated chambers with intensive air circulation. Dimensional control by means of using the hot-flue, is achieved when the material is being guided by rollers and in sieve band dryer. The material is transported in loose and relaxed form by means of air-permeable sieve and dried by circulation of hot air [3].

Pre-drying heaters can be done with IR-radiation which is heated electrically or by gas. These devices give high energy transfers and are normally used for rapid evaporation of water. They are not used for full drying because the temperature of the material is controlled by the evaporation of the water, and this can rise rapidly to ignition if the fabric or material starts to dry completely [3].

After the dyeing process has reached equilibrium, the dye is normally on the exterior zone of the textile fiber. This can bleed out of the textile fiber and because of this, the dye must be fixed into the textile fiber. The fixation of dyes by dry heat (thermofixation) can be also carried out in suitable driers [2]. The heat treatment causes diffusion of dyestuff into the fiber interior during thermosol dyeing by hot air, superheated steam, contact heat or irradiation. In thermosoling, goods must be exposed to constant temperature over the entire width, as temperature fluctuations lead to color differences. The conventional thermosol equipment is shown in figure 22.

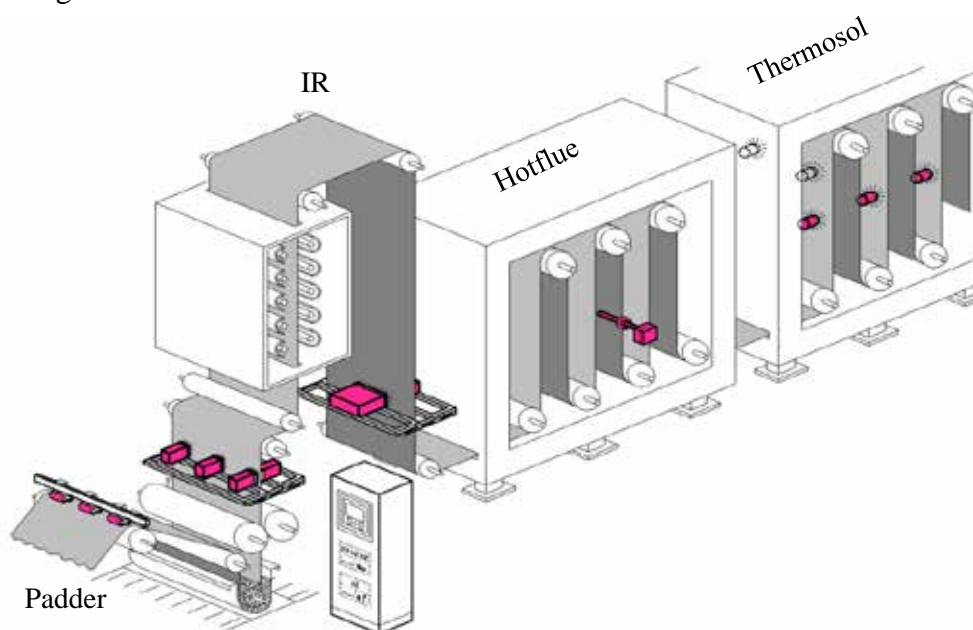


Figure 22: Conventional Thermosol Equipment (Pleva GmbH)

There are two various processes used practically for thermofixation of dyes which are

- drying on the padder followed by thermofixation of dyes and
- drying and fixation in one stage.

Synthetic fibers like polyester, which still holds some amount of dyestuff even after drying, are later heated up above their glass transition temperature. The fabric is normally covered with a dried film of disperse dyes and auxiliary, which is heated to ca. 210-220 °C for 0.5 – 1 min. This causes the dye to diffuse into the interior of the fiber and this makes the dye fixed. Fixation of disperse dyes on polyester fibers as a function of time and temperature can be seen in Figure 23.

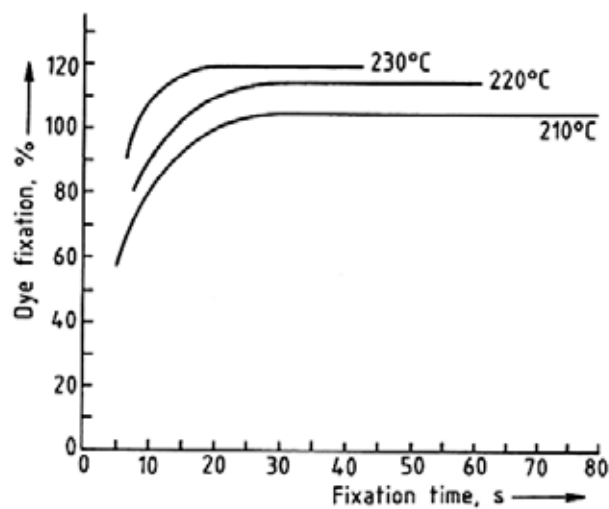


Figure 23: Fixation of disperse dyes on polyester fiber as a function of time and temperature [46]

2.5.1. Convection oven process

Convection (Latin *convectio* = carry along with). This can be explained as heat transfer (temperature equalization) by flow movements according to the buoyancy principle resulting from different density and temperature [2]. This is the most common method of drying using air as the drying medium [2]. Principally, warm air rises in cold air and cold air falls in warm air which allows masses of different temperatures to mix and their temperatures to get equalized by convection [2].

One advantageous use of convective drying is that the conditions under which drying is performed can be varied at will. There are two conditions which are more accepted. These are

- air impingement drying and
- air circulation drying

Air impingement drying technique has an advantage of control to avoid contact on both faces during the drying [2]. For air circulation conditions, the fabric can be vertically placed, which means it alternates up and down through a drying range with circulating hot air. The convection laboratory oven dryer that was used in this project has the fabric placed horizontally. The fabric is clipped on a needle frame (Figure 24) and positioned in the convection oven laboratory dryer (Figure 25) horizontally in the drying area with also circulating hot air.

Diagonal placement of the fabric is also possible where it moves in an up and down zig-zag manner involving double sided (above and below) air flow.



Figure 24: Needle frame (own picture)



Figure 26: convection oven laboratory dryer (own picture)

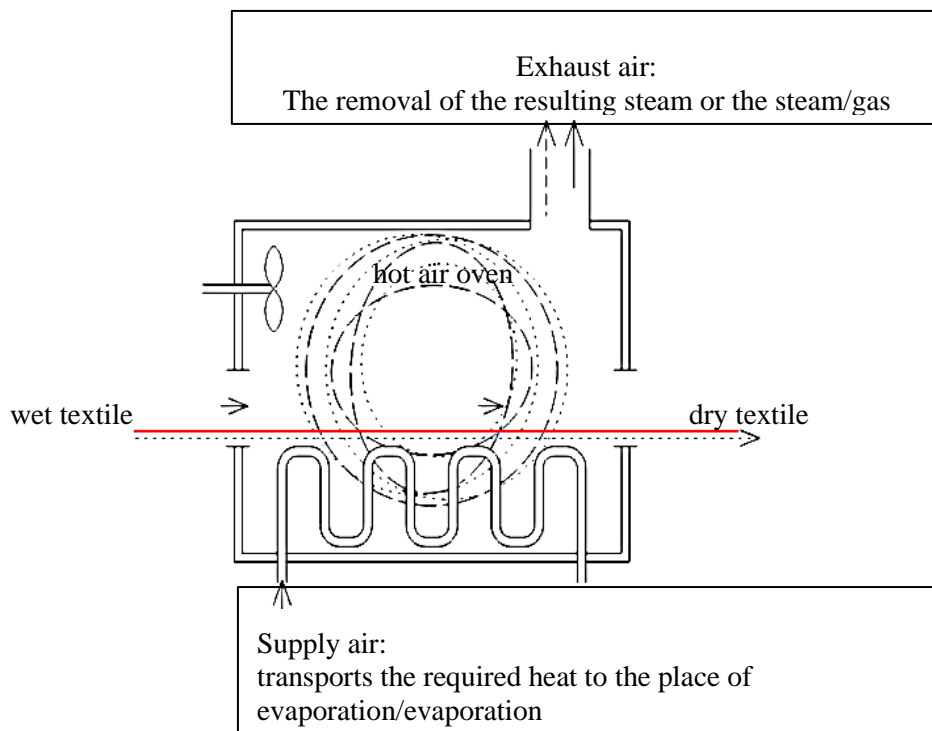


Figure 25: schematic image of convection oven dryer and thermosoling unit (TexPro Consult)

2.5.2. aNIR[®] technology (Advanced near infrared drying) process

Infrared (Latin: infra = below, beneath, and ‘red’). This together translates to be ‘below red’ which is exactly what it means in terms of its frequency [47].

Infrared (IR) radiation is a long wave radiation which cannot be seen with the human eye (Figure 27). The whole IR-spectrum extends from 780 – 400000 nm where only the smaller component of the vibration spectrum between 780 – 3000 nm is of most interest. The transition zone between red in the visible spectrum and IR lies within the range of 700 – 860 nm. Generally, all bodies emit IR-radiation, even in their cold state. The more intense the radiation becomes the hotter the body [2].

Infrared radiation is also called heat radiation because it produces the sense of warmth from non-glowing hot bodies [2]. To produce an artificial IR-radiation like for infrared drying, temperatures above 500 °C are required. IR radiation can set the atomic nucleus in motion by vibrating positively charged atomic particles in molecular associations (dipole moment in cis-form) which are formed as charge carriers [2]. These charge carriers transfer the absorbed radiation energy to the neighboring atoms as so-called collision energy. This is when bodies are been caused to heat up by thermal conduction [2].

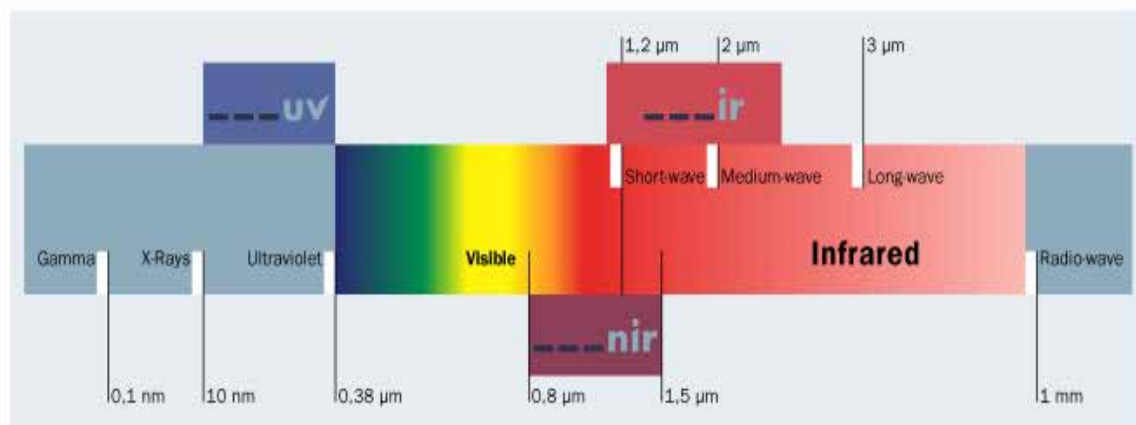


Figure 27: Wavelength spectrum of visible and infrared radiation [49]

In infrared reflection measurement, a continual optical spectrum is evaluated using statistical chemometric procedures like Partial least-squares (PLS). Partial least-squares (PLS) methods for spectral analyses are related to other multivariate calibration methods such as classical least-squares (CLS), Inverse least-squares (ILS), and principal component regression (PCR) methods which have been used often in quantitative spectral analyses.

The PLS method analyzes one chemical component at a time [48]. This PLS method is a statistical method based on Principal Component Regression (PCR) factor analysis, which is particularly suited to quantitative evaluation of the NIR spectra with heavily overlapping absorption bands [2]. The bands are based on the molecular vibrations of the chemical components and can be identified as absorption bands in the infrared spectrum. Using an infrared drying technology, the IR-radiation is used by preference [2]. This technology enables the application of highest energy quantities and densities (up to 1.5 MW/m²) on a broad range of different substrates [49] (Figure 28). The radiation emitted is referred to as the emission, and the radiation, converted into another form of energy when the material which is penetrated, is referred to as absorption [2]. AdvancedNIR[®] radiation with peak wavelengths is in the range of 0.78 – 1.5 μm [49].

aNIR[®] spectrum contains approx. 50% (0.38 – 0.78 μm) visible light and 0.2% UV-radiation (< 0.38 μm) [49].

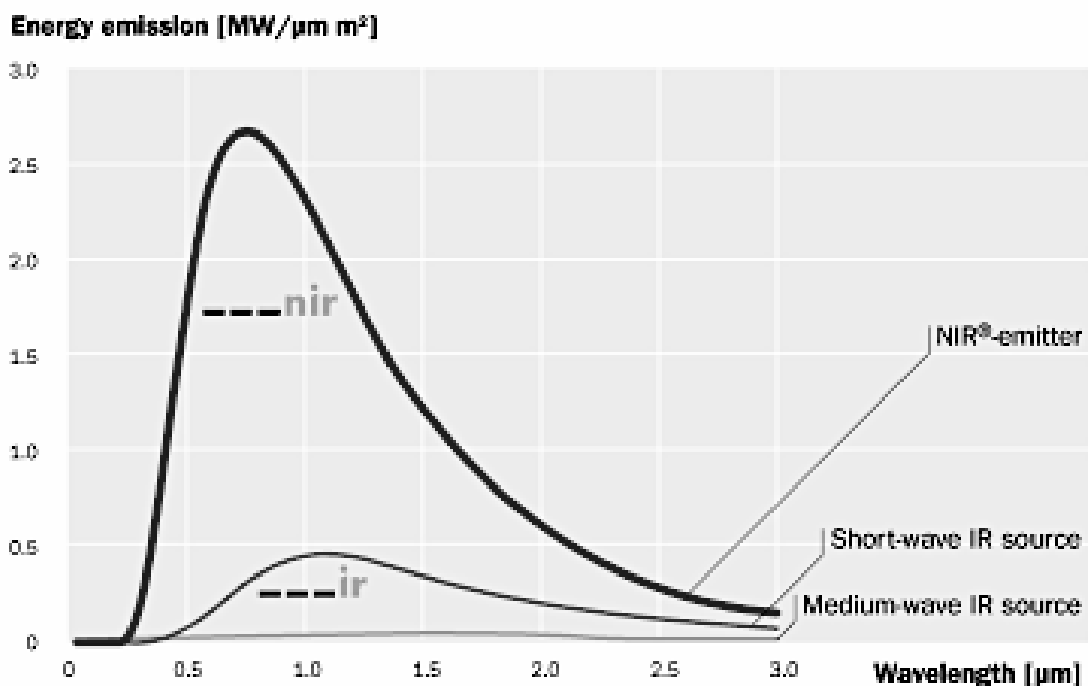


Figure 28: High energy emission of aNIR[®] technology [49]

To achieve a high level of profitability when heating materials, IR radiators with emissions essentially within area around 3000 nm are required. The absorption ratio of textile fabrics like polyester (Figure 29) and water (Figure 30) are also satisfactory in the order of 3000 nm. Radiation temperatures of 500 -800 °C are also preferred [2].

Also, for radiators, the most important components of the IR radiation drier can be categorized:

- Depending in the type of energy used to heat the radiator. Generally, electrical, and gas-heated are used.
- Depending on the temperature of radiator
- Depending on the composition of the radiator
- Depending on the portion of visible light; Emission of light starts at temperature above 500°C [49].

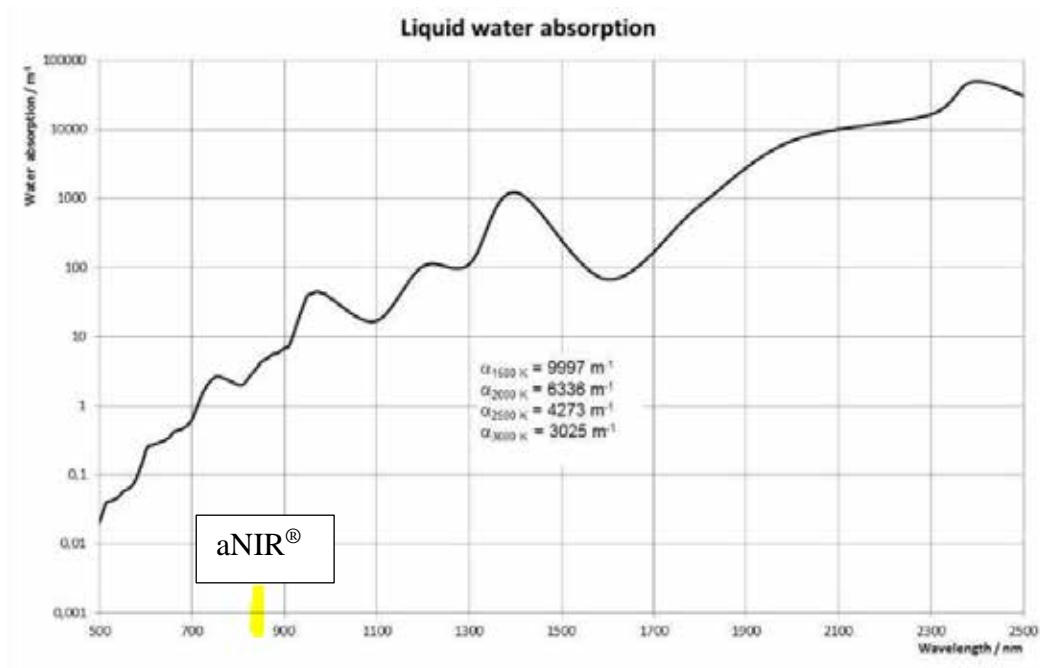


Figure 30: Water absorption spectrum [51]

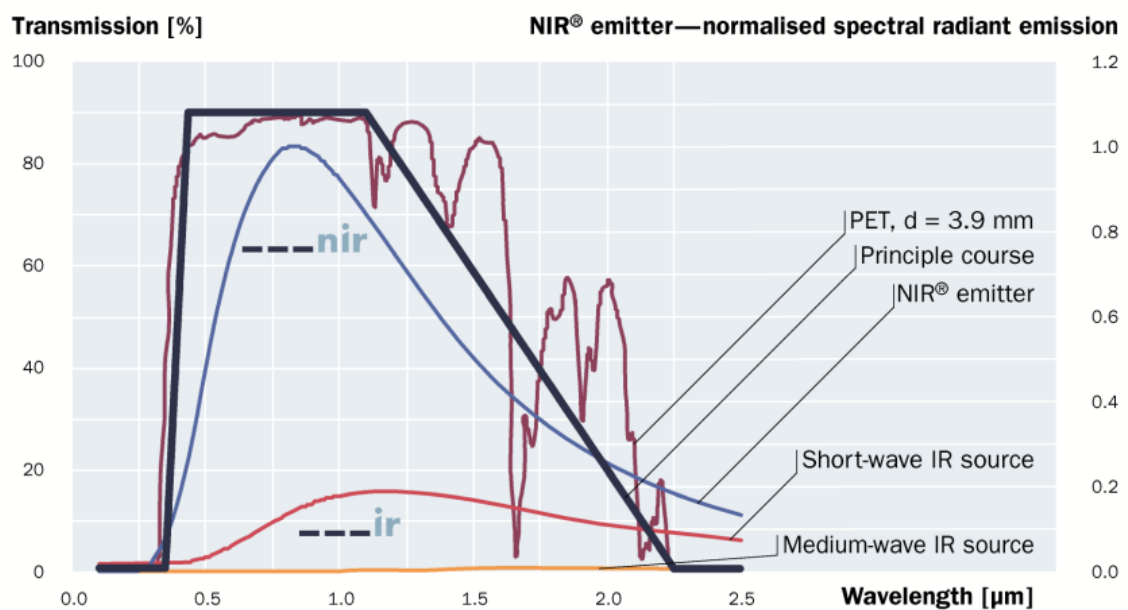


Figure 29: Absorption/Transmission of NIR Radiation in PET Applications [49]

There are several IR emitters in the industry which are used as IR driers. One example is the high temperature quartz tube emitter with an electrically heated tungsten spiral from Adphos Digital Printing GmbH. The advantages of adphosNIR[®] emitters are, they have

- High temperatures of 3,000 – 3,200 K
- long average lifetime of 5,000 h
- compact build
- response time in seconds
- Additional quartz glass inlet can be used as a MIR-blocker [49]

aNIR[®] radiation are different when compared to other drying technologies like IR-radiation in conventional drying, Hot air in conventional drying, etc. Figure 31 [49].

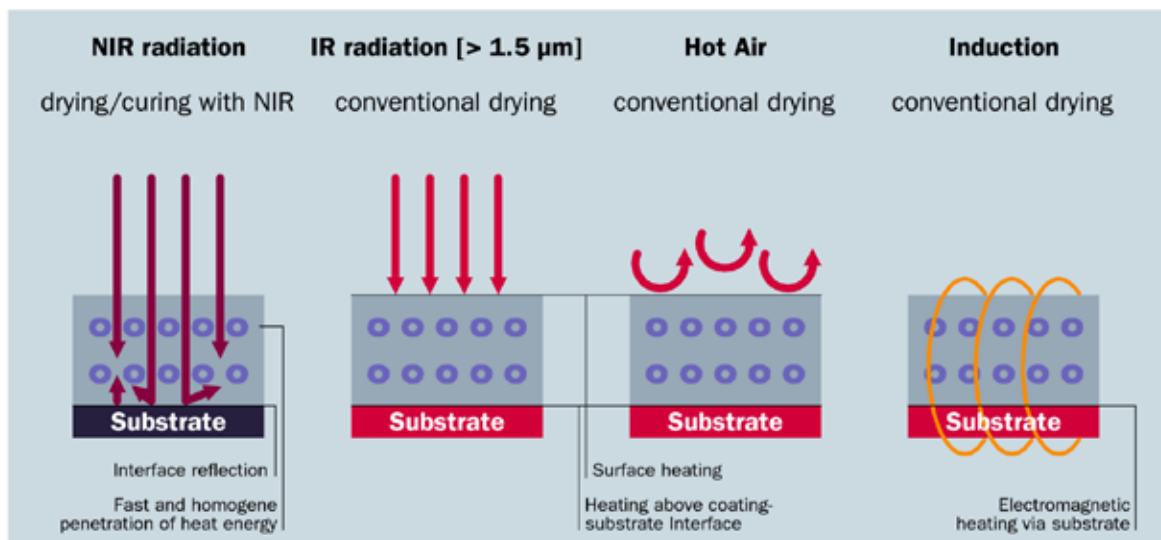


Figure 31: Differences between aNIR[®] radiation and other drying technologies [49]

The central component for most IR dryers needs three structural subsystems, which are the reflectors/emitter arrangement, control system and cooling system [49]. Adphos Digital Printing GmbH have an emitter arrangement of exchangeable plug-in emitters with power output of 0.8 up to 6 kW. The aNIR[®] emitters are not able to generate the energy alone, thus emitters and reflector geometry needs to optimize to gain a brilliant radiation homogeneity and energy densities as well as big, sharply defined heated areas for optimized energy disposition [49].

This system should have a control system which can identify emitter failures, control individual heating zones, integrate temperature controls and other additional interfaces for additional sensors. The cooling systems can be water cooled or air cooled depending on preference.

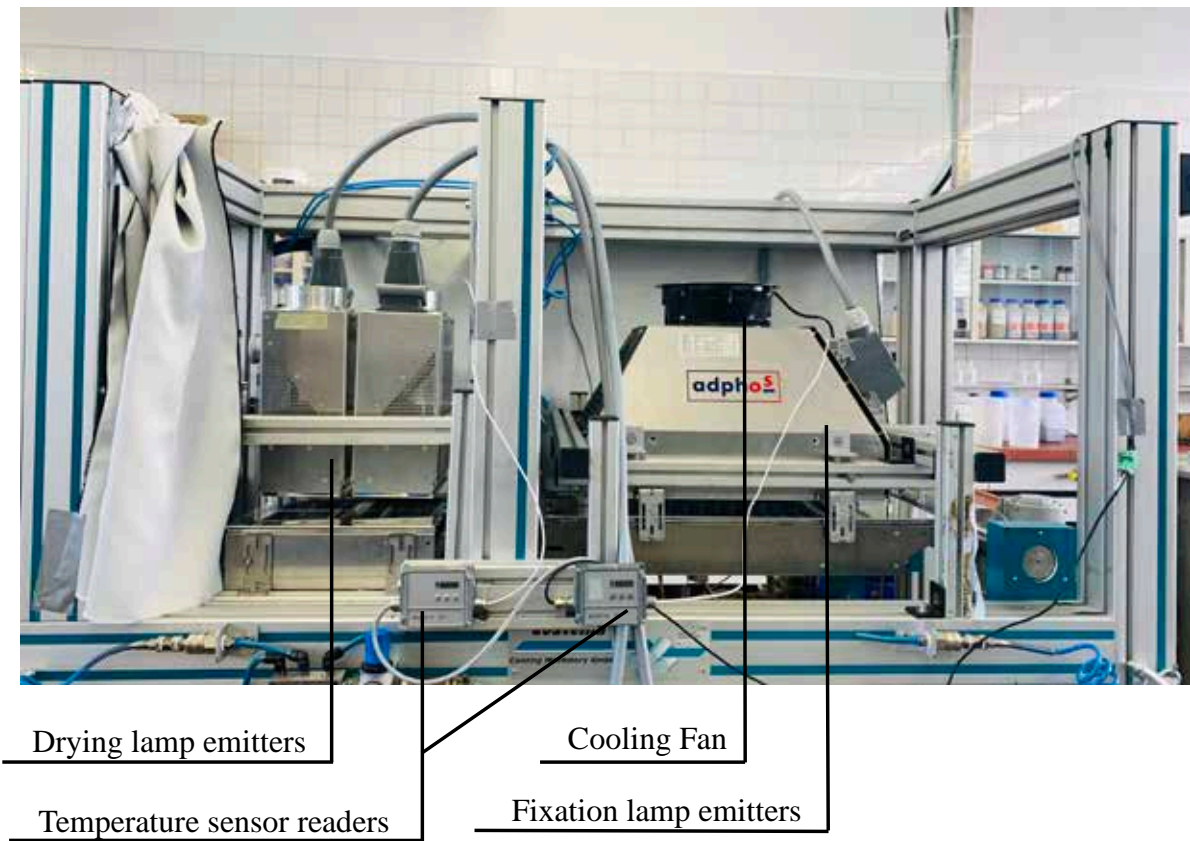


Figure 32: aNIR[®] technology setup (own picture)

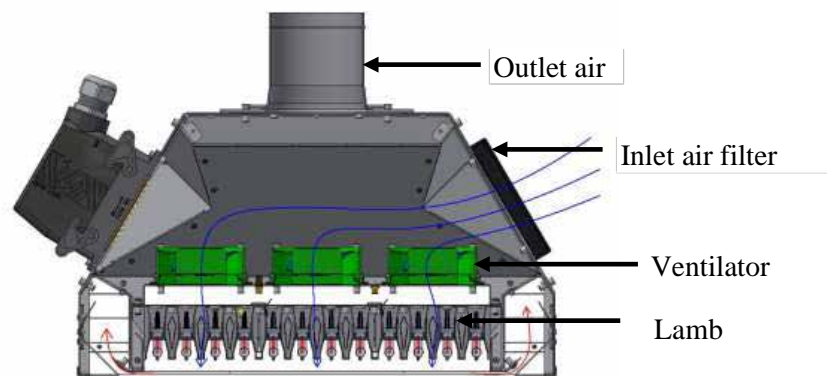


Figure 33: Module of aNIR[®] lamp emitter (air cooled) [52]

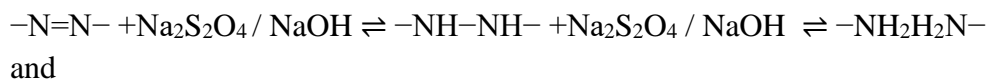
2.6. Aftertreatment

Aftertreatment is done to improve fastness properties of dyed fibers or fabrics. This process is done after dyeing goods to remove excess dyes on the surface of the fabric. For light or medium polyester dyeing's, the material needs to be only rinsed thoroughly or soaped after the dyeing process. For the dark shades, unfixed dye particles are firmly attached to the fiber surface which decrease the fastness. The solution for this is to subject it to reductive alkaline clearing after dyeing where mostly hydrosulphite (sodium dithionite) is used in the presence of a dispersing washing agent.

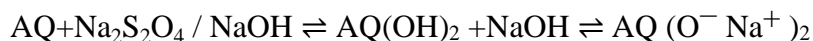
2.6.1. Reductive clearing

After a dyeing process is completed, it must go through a main last stage named the reductive clearing. Most dye molecules of the disperse dyes are dissolved into the fabric or the matrix of the polymer but some minute amounts end up staying on the surface of the fibers. Most of these dyes can be removed during laundering or mechanical action or by a reduction clearing process where dispersants and strong reducing agents (e.g., $\text{Na}_2\text{S}_2\text{O}_4/\text{NaOH}$ at 70°C) are involved. The latter, which is removing the excess dyed molecules on the surface of the fibers is the best method.

There are two different principles for removing disperse dyes if the usage of reducing agent is involved. These are



and



Source: [3]

A reductive cleavage of azo groups presents, forms part of the chromophore in disperse dyes, lead to the destruction of the azo chromophore and the formation of amino components that exhibit higher solubility in the treatment bath [3]. Whereas the anthraquinoid chromophore is stable against the action of reducing agents. Nevertheless, it is reduced by chemicals into the corresponding dihydroxy form, which dissociates in the alkaline bath as a leuco di-anion [1]. Care must be taking in consideration to avoid the reoxidation of the hydrazo form or the leuco di-anion occur on the surface of the fiber before releasing the reductive rinse bath.

2.6.2. Washing with special dispersing agents

There are several requirements of an ideal washing off agent. Typically, it should be compatible to many recipes being effective over a broad PH range [53]. Polymeric and non-foaming should be a feature it should have where it can aid, remove unfixed dyestuff. Effective special dispersing agents need be able to prevent oligomers and machine cleaning after dispersing dyeing of polyester [50].

An example of such is DyStars[®] Sera[®] Sperse C-SN which is a chelating and dispersing agent. As an anionic auxiliary, it is used as a universal soaping agent and during a dyeing process in the dye pad liquor.

3.0. Materials

The materials used in this study are mainly disperse dyes, polyester fiber, auxiliary for dyeing and aftertreatment (reduction clearing) and color fastness.

The disperse dyes used are selected from DyStar[®] which are high energy disperse dyes Dianix[®] XF2 and medium energy disperse dyes Dianix[®] CC.

Dianix[®] XF2 disperse dyes are dyes for the highest levels of wash- and wet-fastness [51]. They are highly productive dyes, especially on polyester/cellulose blends, through alkaline clearing of unfixed dye, eliminating the need for reduction clearing for most of the dyes.

Dianix[®] CC disperse dyes are compactible range of economical, medium-energy dyes for rapid, reliable exhaust and continuous dyeing of polyester and its blends with other fibers. The attribute excellent Right-First-Time performance through compatibility of dyes. They have excellent build-up and economical dye recipes and are good all-round colour fastness [51].

The selected dyes used were

Dianix[®] Rubine XF2

Dianix[®] Navy XF2

Dianix[®] Black 300% XF2

Dianix[®] Yellow Brown XF2

Dianix[®] Rubine CC

Dianix[®] Navy CC

Dianix[®] Black CC-R

Dianix[®] Yellow Brown CC

Table 1: recommended auxiliaries for this dyeing process (DyStar Group)

Auxiliary	Classification	Primary chemical group
Sera [®] Wet C-ANS	Wetting and deaerating agent	Alkylsulfonate, Fattyalcohol polyglycolether
Sera [®] Print M-DP	Anti-migration agent	Polyacrylate
Sera [®] Sperse C-SN	Dispersing agent	Sodium polyacrylate

The polyester fabric which was used for this project are in two sizes: one for conventional process and another for aNIR[®] technology process. The polyester fabric for conventional process is made with a 25 cm width fabric and 50 m long rolled and the polyester fabric for aNIR[®] technology has a width of 10 cm and 50 m long rolled (Figure 37). The fabric weighs ca.170 g/m². The fabric has a glossy surface at one side which was standardised as the face of the fabric and the non-glossy part as the back. The warp of the fabric has 78 dtex and 357 dtex weft.



Figure 34: Polyester fiber: Conventional process;left aNIR[®] Technology process; right (own picture)

The standard recipe used for both conventional process and aNIR[®] technology process is as follows:

Standard recipe

Disperse dyestuff	x	[g/l]
Sera [®] Wet C-ANS	2	[g/l]
Sera [®] Print M-DP	10	[g/l]

pH 5.5: adjusted with acetic acid

Dissolving temperature	40-60	[°C]
pad liquor pick-up	68	[%]

The dyestuff concentration (x) of pale, medium and dark shades of the dyestuff was calculated according to the standard depth of the DyStar® 's Dianix® shadecard published in 2020.

Formular for calculating the concentration of disperse dye shades x [g/l]

$$x \text{ [g/l]} = \frac{\text{standard depth}}{\text{pick up}} \times 10$$

The value of the standard depth must be ratioed to develop the shades of pale, medium and dark. In pattern sheet card, the concentration of dyestuff is only mentioned for 1/1 standard depth.

Pale – 1/6 of standard depth

Medium – 1/1 of standard depth [51]

Dark – 2/1 of standard depth

Standard depth for Dianix® Navy CC/XF2 and Dianix Black CC-R/300%XF2 is called NavyBlue and Black. In pattern sheet card, the concentration of dyestuff is only mentioned for NavyBlue and Black.

Pale – 1/6 of NavyBlue and black

Medium – 1/2 of NavyBlue and black

Dark – NavyBlue and Black [51]

After calculating all dyestuff concentrations according to their standard depth in pale, medium and dark shades are shown in Table 2.

Table 2: Dianix® disperse dyes concentration in shades [g/l]

Dianix® disperse dyes	Shades [g/l]		
Rubine XF2	Pale	-	2.5
	Medium	-	15
	Dark	-	30
Navy XF2	Pale	-	5.8
	Medium	-	17.5
	Dark	-	35
Yellow Brown XF2	Pale	-	3.3
	Medium	-	20
	Dark	-	40
Black 300% XF2	Pale	-	9.3
	Medium	-	28
	Dark	-	56
Rubine CC	Pale	-	1.5
	Medium	-	9
	Dark	-	18
Navy CC	Pale	-	3
	Medium	-	9
	Dark	-	18
Black CC-R	Pale	-	7
	Medium	-	21
	Dark	-	43
Yellow Brown CC	Pale	-	1.8
	Medium	-	11
	Dark	-	22

A combination of 3 Dianix[®] CC disperse dyes, and 3 Dianix[®]XF2 disperse dyes were used to produce trichrome brown sample.

Dianix[®] Trichrome Brown Recipe

Dianix [®] Navy CC / XF2	2 [g/l]
Dianix [®] Rubine CC / XF2	4 [g/l]
Dianix [®] Yellow Brown CC / XF2	8 [g/l]
Sera [®] Wet C-ANS	2 [g/l]
Sera [®] Print M-DP	10[g/l]
pH 5.5: adjusted with acetic acid	
Dissolving temperature	40-60 [°C]
pad liquor pick-up	68 [%]

Preparation for standard process

The standard process used is a discontinuous process involving foulard unconnected from the convection oven dryer and thermosoling (Figure 38). The standard process and the selected parameters include

Step by step process

- Firstly, the polyester fabric sample is cut with a dimension of 25 cm by 20 cm.
- The dye liquor is prepared using the standard recipe according to the specific Dianix[®] disperse dyes.
- The dye liquor is filled in the foulard; combination of dipping bath tray and padder rollers.
- The foulard speed should be set the same as the running speed of the aNIR[®] technology process.
- Polyester fabric sample is then soaked in the dye liquor in the dipping bath tray and passed through the padder rollers.
- The dyed polyester fabric sample is now stretched tightly onto a frame and carried out to the convection oven dryer.

Convection oven drying parameters

- 100 °C oven temperature
- 700 rpm fan speed
- Time: 300 [s]

Thermosoling

- Temperature for Dianix[®] CC disperse dyes 210 °C
- Temperature for Dianix[®] XF2 disperse dyes 220 °C
- 2160 rpm fan speed
- 60 [s]



Figure 36: Image of foulard and convection oven dryer and thermosoling unit. (Own picture)

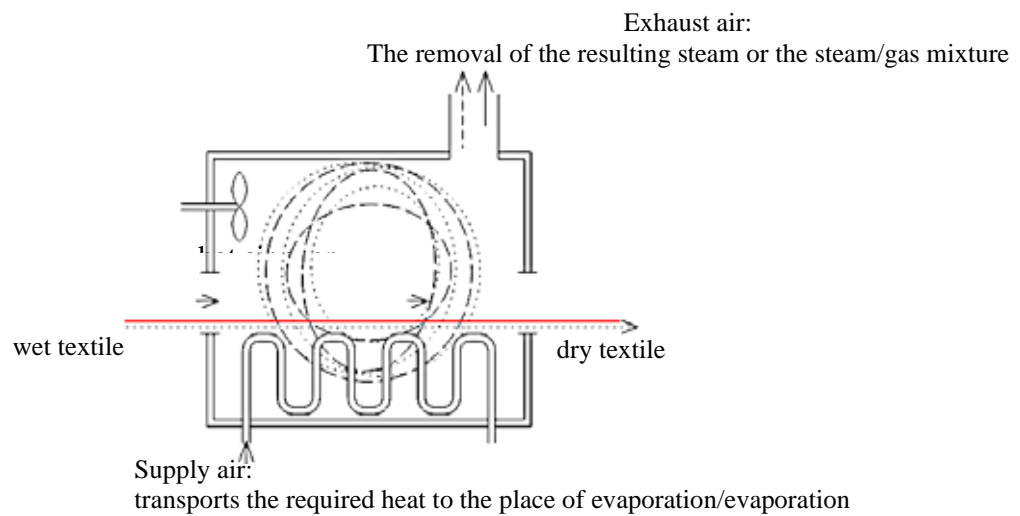


Figure 35: Schematic drawing of convection oven dryer and thermosoling unit (TexPro Consult).

Preparation for aNIR[®] technology process

The aNIR[®] technology uses a continuous roll to roll process involving a foulard the aNIR[®] drying units connected to the aNIR[®] fixation unit (Figure 40). The drying and fixation emitters have cooling fans above and temperature sensor placed at the end of every unit (drying and fixation) to read the surface temperature of the fabric. At a temperature above 80°C which is called the wet bulb temperature of the fabric, is said to be the point at which the dyed fabric is considered to be dry and these temperature sensor help in achieving this goal. All lamp emitters are positioned above reflectors for even irradiation.

The aNIR[®] drying unit involves two set of lamp emitters installed besides each other. Each drying lamp emitter measures 84 mm (both 168 mm) width and 200 mm long (figure 42) and contains two lamps in each of them. The fixation emitter is measured 360 mm width and 500 mm long (Figure 43).



Figure 37: aNIR[®] technology setup (continues process) (own picture)

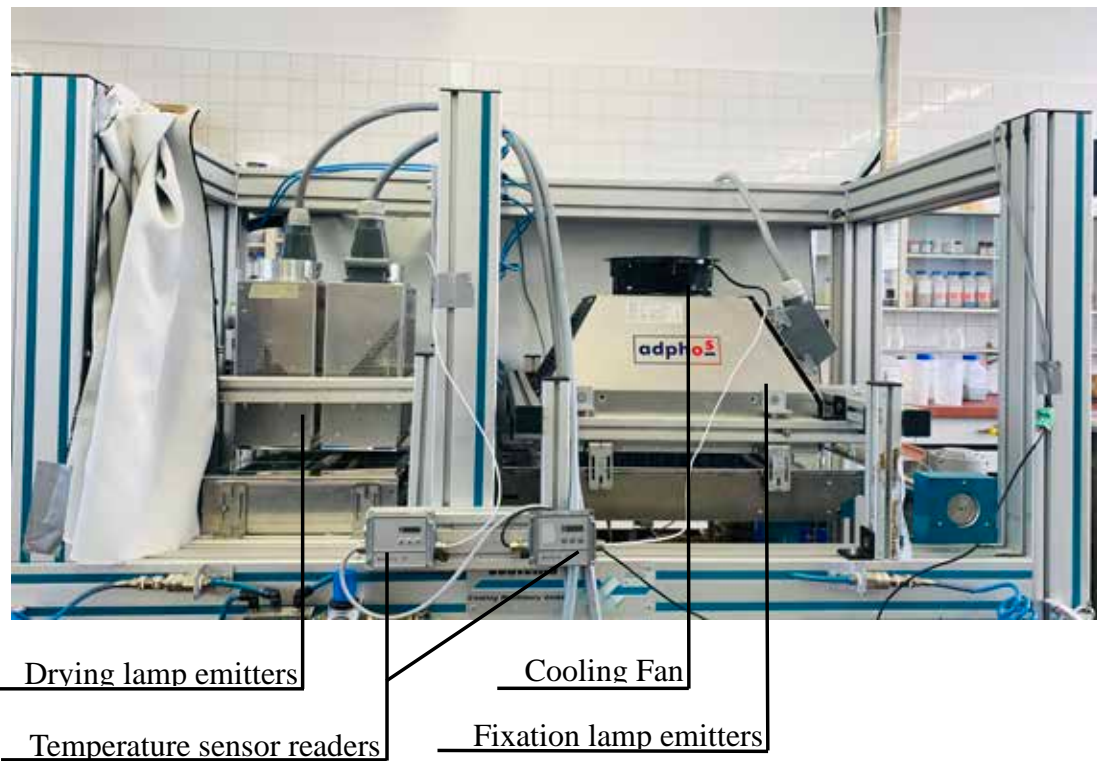


Figure 38: aNIR® technology setup (Drying and Fixation emitters) (own picture)

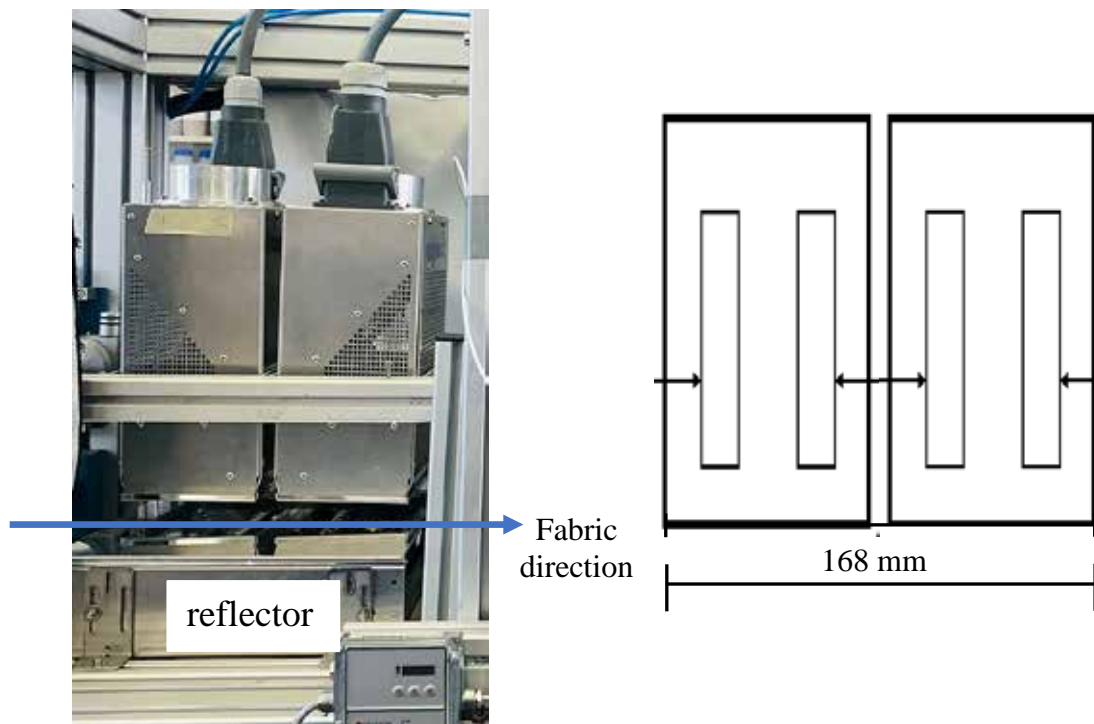


Figure 39: Image and schematic drawing of drying aNIR® lamp emitters (own picture)

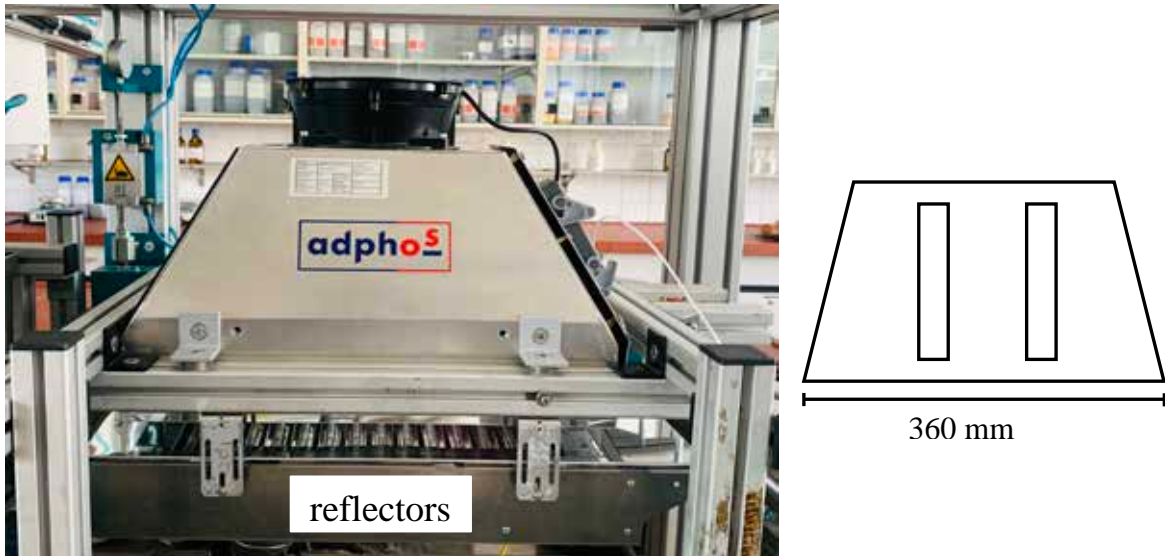


Figure 40: image and schematic drawing of fixation aNIR® lamp emitters (own picture)

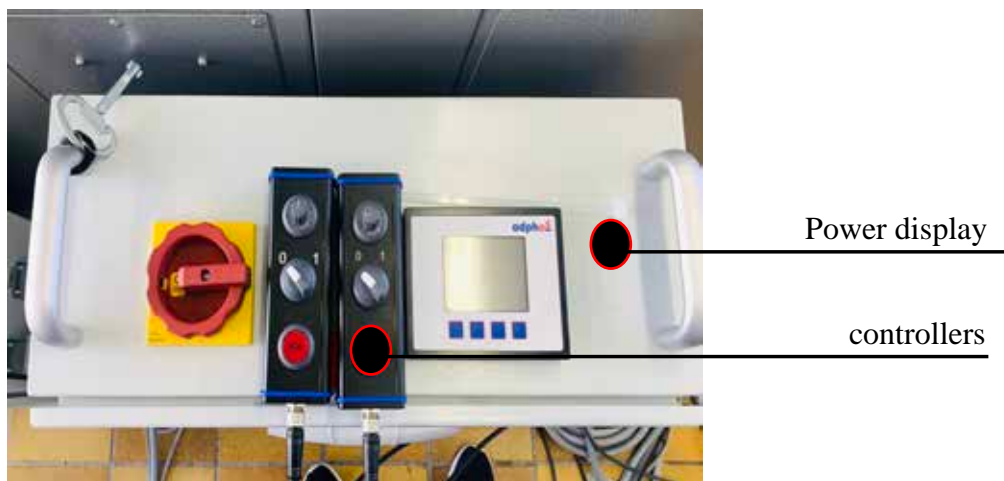


Figure 41: image of controllers and power display (Own picture)

Step by step process

- Firstly, the polyester fabric sample is positioned on a feeder roller through the opened foulard, between both the drying and fixation emitters and onto a bobbin.
- The dye liquor is prepared using the standard recipe according to the specific Dianix® disperse dyes.
- The dye liquor is then filled in the foulard
- The foulard speed is set to run 4m/m.
- The aNIR® machine is then started by switching on with the controllers set at low power.

- Final adjustments of the polyester fabric are made with the positioning in the foulard and under the lamp emitters.
- The foulard is switched on and the lamp emitters turned up for a run
- The polyester fabric for aNIR[®] technology is run through the dye liquor in the dipping bath and passes through the squeeze rollers and proceeds under the aNIR[®] drying and fixation emitters
- The temperature/power controllers are turned until the recommended temperature for the individual dyestuff is reached

aNIR[®] drying lamp emitter parameters

drying temperature - 90 °C

power of emitter - x % where 100% = 8 kW (maximum)

aNIR[®] fixation lamp emitter parameters

- Temperature for Dianix[®] CC disperse dyes 210 °C
- Temperature for Dianix[®] XF2 disperse dyes 220 °C
- power of emitter - x % where 100% = 9.6 kW (maximum)

Table 3: Dwell time (green = standard speeds)

Machine Speed [m/min] / [mm/s]	Dwell Times [s]	
	Drying phase	Thermofixation
1 / 16,7	10,1	21,6
2 / 33,4	5,1	10,8
3 / 50,0	3,4	7,2
4 / 66,7	2,5	5,4
5 / 83,3	2,0	4,3

Formula for calculating the dwell time of the polyester fabric under the emitters is as follows.

$$\text{dwell time [s]} = \frac{\text{distance [mm]}}{\text{speed [mm/s]}}$$

Standardised After Treatment (reduction clearing)

Recommended: NaOH 50% 6 [ml/l]

Available: NaOH 33%

Therefore $\frac{6 \text{ [ml/l]} \times 50\%}{33\%} = 9.1 \text{ [ml/l]}$

NaOH 33% 9.1 [ml/l]

Sodium Dithionite 7 [g/l]

Sera[®] Sperse C-SN New 1-2 [g/l]

15 min at 60 °C

Hot rinsing

5 min at 60 °C

Neutralisation

Acetic acid 5 [ml/l]

Cold rinsing

5 min

Spinning dryer

5 min

Air drying overnight

Firstly, a conventional process sample dyeing was made to test out the materials and machine setup. In total 8 samples were made: 6 dyed samples and 2 blank samples without dye and thermofixed with convection process under 200/210 °C. The dye colour selected was Dianix[®] Black CC-R and Dianix[®] Black 300% XF2 disperse dyes.

The dye concentration used are as follows

Dianix[®] Black CC-R (trial 1)

Pale - 1 [g/l]

Medium -10 [g/l]

Dark -20 [g/l]

Dianix[®] Black 300% XF2 (trial 1)

Pale -15 [g/l]

Medium -30 [g/l]

Dark -60 [g/l]

Blank sample (without dye) 200 °C / 215 °C

Sera® Wet C-ANS 2 [g/l]

Sera® Print M-DP 10 [g/l]

pH 5.5: adjusted with acetic acid

Dissolving temperature 40-60 [°C]

pad liquor pick-up 68 [%]

After that, proceedings were made with the standard convection oven drying and Thermosoling. The standard After Treatment (reduction clearing) was done after dyeing. A second sample test was made by both convection process and aNIR® technology with Dianix® Black CC-R and in similar dye concentration.

Dianix CC-R new Black (trial 2)**Recipe****Convection process**

Dianix CC-R new Black 30 [g/l]

Sera® Wet C-ANS 2 [g/l]

Sera® Print M-DP 10 [g/l]

pH 5.5: adjusted with acetic acid

Dissolving temperature 40-60 [°C]

pad liquor pick-up 68 [%]

foulard speed 4 [m/min]

Standard convection oven drying and Thermosoling

Standard After Treatment (reduction clearing)

aNIR® technology process

Dianix Black CC-R new	30	[g/l]
Sera® Wet C-ANS	2	[g/l]
Sera® Print M-DP	10	[g/l]

pH 5.5: adjusted with acetic acid

Dissolving temperature	40-60	[°C]
pad liquor pick-up	68	[%]
foulard speed	4	[m/min]

Drying temperature	90	[°C]
Power of emitter	100	[%]
Dwell time	2.5	[s]

Fixation temperature	210	[°C]
Power of emitter	85	[%]
Dwell time	5.4	[s]

After dyeing the standard after treatment (reduction clearing) is done. After the second test sample, the next test involved the same recipe but a change in the antimigration agent to check the effect of different products and concentrations.

Dianix® Black CC-R new (Trial 3)

Dianix® Black CC-R new	30	[g/l]
Sera® Wet C-ANS	2	[g/l]
Sera® Gal M-SPR	7	[g/l]

pH 5.5: adjusted with acetic acid

Dissolving temperature	40-60	[°C]
pad liquor pick-up	68	[%]
foulard speed	4	[m/min]

Drying temperature	90-95	[°C]
Power of emitter	100	[%]
Dwell time	2.5	[s]

Fixation temperature	205-210	[°C]
Power of emitter	76	[%]
Dwell time	5.4	[s]

After dyeing the standard after treatment (reduction clearing) is done.

Dianix® Black CC-R new (Trial 3)

Dianix Black CC-R new	30	[g/l]
Sera® Wet C-ANS	2	[g/l]
Sera® Gal M-SPR	30	[g/l] (maximum)

pH 5.5: adjusted with acetic acid

Dissolving temperature	40-60	[°C]
pad liquor pick-up	68	[%]
foulard speed	4	[m/min]

Drying temperature	90-98	[°C]
Power of emitter	100	[%]
Dwell time	2.5	[s]

Fixation temperature	209-213	[°C]
Power of emitter	77	[%]
Dwell time	5.4	[s]

After dyeing the standard after treatment (reduction clearing) is done.

The next set of dyeing was to dye all Dianix®XF2 disperse dyes by both convection and aNIR® technology process since specific parameters were established.

Convection process

Standard recipe

Dianix® XF2	x	[g/l]
Sera® Wet C-ANS	2	[g/l]
Sera® Print M-DP	10	[g/l]

pH 5.5: adjusted with acetic acid

Dissolving temperature	40-60	[°C]
pad liquor pick-up	68	[%]
foulard speed	4	[m/min]

Standard convection oven drying and Thermosoling
Standard After Treatment (reduction clearing)

aNIR® technology process

Dianix® Rubine XF2 x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature	85-90	[°C]
Power of emitter	87	[%]
Dwell time	3.4	[s]

Fixation temperature	218-222	[°C]
Power of emitter	100	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature	90-100	[°C]
Power of emitter	82	[%]
Dwell time	3.4	[s]

Fixation temperature	218-225	[°C]
Power of emitter	87	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature	88-94	[°C]
Power of emitter	75	[%]
Dwell time	3.4	[s]

Fixation temperature	220-226	[°C]
Power of emitter	82	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Yellow Brown XF2 x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature 90-94 [°C]

Power of emitter 92 [%]

Dwell time 3.4 [s]

Fixation temperature 215-221 [°C]

Power of emitter 100 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature 89-93 [°C]

Power of emitter 80 [%]

Dwell time 3.4 [s]

Fixation temperature 219-224 [°C]

Power of emitter 88 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature 90-93 [°C]

Power of emitter 75 [%]

Dwell time 3.4 [s]

Fixation temperature 218-224 [°C]

Power of emitter 82 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Navy XF2 x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature 89-95 [°C]

Power of emitter 85 [%]

Dwell time 3.4 [s]

Fixation temperature 204-212 [°C]

Power of emitter 83 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature 89-93 [°C]

Power of emitter 75 [%]

Dwell time 3.4 [s]

Fixation temperature 208-213 [°C]

Power of emitter 72 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature 92-96 [°C]

Power of emitter 73 [%]

Dwell time 3.4 [s]

Fixation temperature 201-211 [°C]

Power of emitter 71 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Black 300% XF2 x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature	89-98	[°C]
Power of emitter	81	[%]
Dwell time	3.4	[s]

Fixation temperature	210-225	[°C]
Power of emitter	86	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature	95-107	[°C]
Power of emitter	76	[%]
Dwell time	3.4	[s]

Fixation temperature	216-219	[°C]
Power of emitter	76	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature	102-109	[°C]
Power of emitter	72	[%]
Dwell time	3.4	[s]

Fixation temperature	220-221	[°C]
Power of emitter	73	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

There was an observation of a migration effect and a difference of color yield in the face and back of the dyed fabric. This influenced a proceeding of dyeing with Dianix[®] Trichrome Brown using several combinations of ambient air dried, convection oven dry and aNIR[®] technology process. To minimize migration effects during intermediate drying, ambient air was used. Ambient air dried is done by laying down the dyed sample on a stretch of a table and allowed to dry by ambient air. The recipe used was the Dianix[®] Trichrome CC Brown Recipe.

First sample combination (Ambient air dried with convection oven thermosoling)

Temperature for Dianix[®] CC disperse dyes 210 °C

2160 rpm fan speed

60 [s]

After dyeing the standard after treatment (reduction clearing) is done

Second sample combination (Ambient air dried with aNIR[®] fixation)

Fixation temperature 207-212 [°C]

Power of emitter 82 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Third sample combination (aNIR[®] drying technology with convection oven thermosoling)

aNIR Drying temperature 95-95 [°C]

Power of emitter 77 [%]

Dwell time 3.4 [s]

Convection oven themosoling

Dianix[®] CC disperse dyes 210 °C

2160 rpm fan speed

60 [s]

After dyeing the standard after treatment (reduction clearing) is done

Fourth sample combination (aNIR® technology process)

Drying temperature	92-95	[°C]
Power of emitter	77	[%]
Dwell time	3.4	[s]
Fixation temperature	209-212	[°C]
Power of emitter	78	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

After this combination of drying and fixation methods were completed, another process of minimal application was produced to find out if there is a change in the migration effect on the dyed fabric. The minimal application used was the Rotating Atomizer (spray) technology produced on a RotaDyer lab machine at the technical centre of WEKO/RotaSpray. This process was done in a batchwise process where the fabrics were dyed with minimal application and transferred to combination of drying and fixation processes. Dianix® Trichrome CC/XF2 Brown disperse dye recipe was used with a pick up adjustment to obtain the same amount of dyestuff concentration used in the aNIR® technology process.

Step by Step

- Firstly, the dye liquor solution is prepared according to the standard depth
- The liquor pick up was calculated corresponding to the aNIR® technology.
- The polyester fabric is then positioned in the RotaDyer Lab machine to be ready for dyeing
- The dye liquor is then transferred to the RotaDyer Lab machine system through its dye feeder tray
- The RotaDyer Lab machine is then turned on to get the process running
- The dye liquor runs through the system and comes out through a nozzle above of a rotating atomizer
- The dye liquor sprouts out of the nozzle, on the rotating atomizer which sprays the dye liquor on the fabric.

Dianix® Trichrome Brown CC recipe (Rotating Atomizer (spray) technology)

Dianix® Navy CC / XF2	4.2	[g/l]
Dianix® Rubine CC / XF2	8.4	[g/l]
Dianix® Yellow Brown CC / XF2	16.7	[g/l]

Sera® Gal M-SPR 3 [g/l]

pH 5.5: adjusted with acetic acid

Dissolving temperature 40-60 [°C]

RotaSpray liquor temperature 22 – 29[°C]

Liquor pick-up 32.6 %

After dyeing with the Rotating Atomizer (spray) technology these drying and fixation combinations were done

First sample combination of Rotating Atomizer (spray) technology (Ambient air dried with convection oven thermosoling)

Temperature for Dianix® CC disperse dyes 210 °C

Temperature for Dianix® XF2 disperse dyes 220 °C

2160 rpm fan speed

60 [s]

After dyeing the standard after treatment (reduction clearing) is done

Second sample combination of Rotating Atomizer (spray) technology (Ambient air dried with aNIR® fixation)

Temperature for Dianix® CC disperse dyes 210 °C

Fixation temperature 214-216 [°C]

Power of emitter 90 [%]

Dwell time 5,4 [s]

After dyeing the standard after treatment (reduction clearing) is done

Temperature for Dianix® XF2 disperse dyes 220 °C

Fixation temperature 224-228 [°C]

Power of emitter 91 [%]

Dwell time 5,4 [s]

After dyeing the standard after treatment (reduction clearing) is done

Third sample of Rotating Atomizer (spray) technology combination (aNIR[®] technology process)

Temperature for Dianix[®] CC disperse dyes 210 °C

Drying temperature	98-100	[°C]
Power of emitter	60	[%]
Dwell time	2,5	[s]

Fixation temperature	214-216	[°C]
Power of emitter	90	[%]
Dwell time	5,4	[s]

After dyeing the standard after treatment (reduction clearing) is done

Temperature for Dianix[®] XF2 disperse dyes 220 °C

Drying temperature	100-104	[°C]
Power of emitter	64	[%]
Dwell time	2,5	[s]
Fixation temperature	224-228	[°C]
Power of emitter	91	[%]
Dwell time	5,4	[s]

After dyeing the standard after treatment (reduction clearing) is done

The migration effect of the fabrics yielding colour differences of the face and back led to a setup change of the aNIR[®] machine. The change was effective only at the drying unit where the drying lamp emitters were placed opposite to each other instead of the earlier adjacent position. This setup omitted the reflectors below the adjacent drying emitters and made the drying lamp emitters radiate to both the face and back of the dyed fabric simultaneously. By this change, the dwell time also reduced making the process faster in the drying phase. This developed into having two setups of the aNIR[®] technology which were setup 1 and setup 2(Figure 42).

Table 4: Dwell time of setup 1 and setup 2 (green = standard speeds)

Machine Speed [m/min] / [mm/s]	Dwell Times [s]		
	Drying phase (setup 1)	Drying phase (setup 2)	thermofixation
1 / 16,7	10,1	5,0	21,6
2 / 33,4	5,1	2,5	10,8
3 / 50,0	3,4	1,7	7,2
4 / 66,7	2,5	1,2	5,4
5 / 83,3	2,0	1	4,3

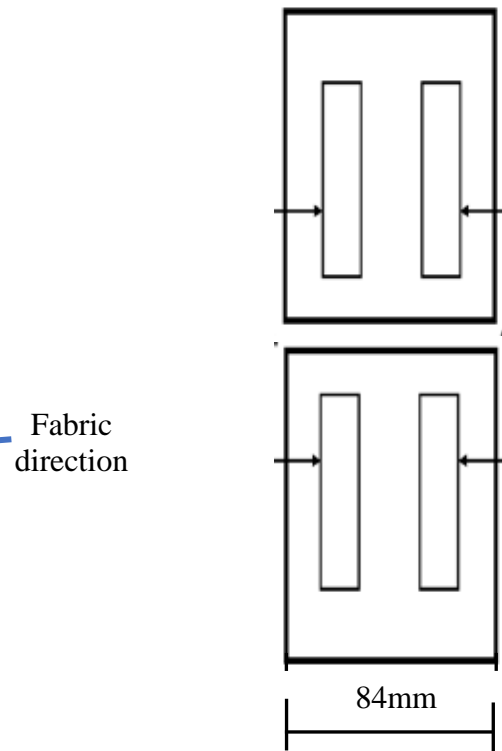
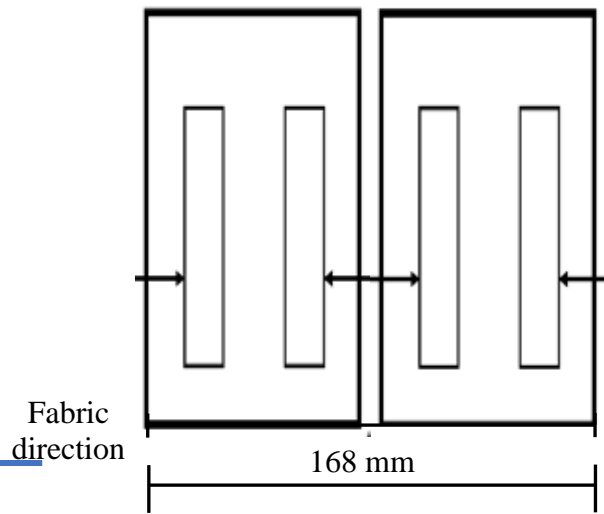


Figure 42: Setup change of aNIR[®] technology (own picture)

The same dyeing process was made with Rotating Atomizer (spray) technology and these drying and fixation combinations were done with setup 2 to check if there are difference in both setup samples.

First sample of Rotating Atomizer (spray) technology combination (aNIR® technology process) setup 2

Temperature for Dianix® CC disperse dyes 210 °C

Drying temperature 107-109 [°C]

Power of emitter 65 [%]

Dwell time 1,2 [s]

Fixation temperature 210-212 [°C]

Power of emitter 83 [%]

Dwell time 5,4 [s]

After dyeing the standard after treatment (reduction clearing) is done

Temperature for Dianix® XF2 disperse dyes 220 °C

Drying temperature 102-104 [°C]

Power of emitter 61 [%]

Dwell time 1,2 [s]

Fixation temperature 223-226 [°C]

Power of emitter 91 [%]

Dwell time 5,4 [s]

After dyeing the standard after treatment (reduction clearing) is done

Since there was a change in the aNIR® technology drying phase, a sample run was also made with standard Dianix® Trichrome CC/XF2 Brown recipe using setup 2.

First sample of aNIR® technology Setup 2

Temperature for Dianix® CC disperse dyes 210 °C

Drying temperature	89-93	[°C]
Power of emitter	80	[%]
Dwell time	1.7	[s]

Fixation temperature 210-211 [°C]

Power of emitter 83 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Temperature for Dianix® XF2 disperse dyes 220 °C

Drying temperature	95-97	[°C]
Power of emitter	80	[%]
Dwell time	1.7	[s]

Fixation temperature 220-224 [°C]

Power of emitter 88 [%]

Dwell time 7.2 [s]

After dyeing, the standard after treatment (reduction clearing) is done

At this point of the process, all samples, combinations, and different machine setups were adjusted and produced to analyze the comparability between padding and Rotating Atomizer (spray) technology (minimal application) and between aNIR® technology and conventional process. Afterward the final samples of Dianix® CC/ XF2 disperse dyes were done with both conventional and aNIR technology processes. The drying phase of Dianix® XF2 disperse dyed fabrics in most of the pale shades needs roughly 18% more energy than the same dye in the dark shades. This shows the absorption dependency on dyestuff concentration. In the thermofixation phase, the same dyestuff shades show the same about 18% difference energy required.

The convection process dries the dyed fabric for 300 seconds and thermofixes it in 60 seconds, the aNIR® technology (1st setup) dries a dyed fabric for 2,5 seconds and thermofixes it in 5,4. In the aNIR® technology (2nd setup) reduces the drying of the fabric in 1,2 seconds and thermofixation in 5,4 seconds.

Standard Recipe for aNIR® technology process setup 2

Dianix®Rubine XF2 x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature 89-93 [°C]

Power of emitter 84 [%]

Dwell time 1.7 [s]

Fixation temperature 218-222 [°C]

Power of emitter 96 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature 89-91 [°C]

Power of emitter 77 [%]

Dwell time 1.7 [s]

Fixation temperature 220-221 [°C]

Power of emitter 88 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature 88-92 [°C]

Power of emitter 77 [%]

Dwell time 1.7 [s]

Fixation temperature 219-223 [°C]

Power of emitter 85 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Navy XF2 x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature	90-94	[°C]
Power of emitter	75	[%]
Dwell time	1.7	[s]

Fixation temperature	219-220	[°C]
Power of emitter	82	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature	89-91	[°C]
Power of emitter	69	[%]
Dwell time	1.7	[s]

Fixation temperature	220-221	[°C]
Power of emitter	76	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature	92-94	[°C]
Power of emitter	68	[%]
Dwell time	1.7	[s]

Fixation temperature	221-224	[°C]
Power of emitter	75	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Black 300% XF2 x [g/l] (Pale, Medium and Dark)
Standard recipe

Pale

Drying temperature	92-94	[°C]
Power of emitter	72	[%]
Dwell time	1.7	[s]

Fixation temperature	220-223	[°C]
Power of emitter	81	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature	89-92	[°C]
Power of emitter	70	[%]
Dwell time	1.7	[s]

Fixation temperature	220-224	[°C]
Power of emitter	76	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature	89-94	[°C]
Power of emitter	68	[%]
Dwell time	1.7	[s]

Fixation temperature	220-222	[°C]
Power of emitter	69	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Yellow Brown XF2 x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature	92-95	[°C]
Power of emitter	86	[%]
Dwell time	1.7	[s]

Fixation temperature	220-225	[°C]
Power of emitter	100	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature	89-92	[°C]
Power of emitter	77	[%]
Dwell time	1.7	[s]

Fixation temperature	221-223	[°C]
Power of emitter	85	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature	91-94	[°C]
Power of emitter	73	[%]
Dwell time	1.7	[s]

Fixation temperature	220-222	[°C]
Power of emitter	78	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Yellow Brown CC x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature 88-90 [°C]

Power of emitter 83 [%]

Dwell time 1.7 [s]

Fixation temperature 206-210 [°C]

Power of emitter 100 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature 89-90 [°C]

Power of emitter 80 [%]

Dwell time 1.7 [s]

Fixation temperature 210-212 [°C]

Power of emitter 92 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature 89-93 [°C]

Power of emitter 76 [%]

Dwell time 1.7 [s]

Fixation temperature 209-212 [°C]

Power of emitter 83 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Navy CC x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature 90-93 [°C]

Power of emitter 80 [%]

Dwell time 1.7 [s]

Fixation temperature 210-213 [°C]

Power of emitter 85 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature 87-90 [°C]

Power of emitter 75 [%]

Dwell time 1.7 [s]

Fixation temperature 210-212 [°C]

Power of emitter 81 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature 89-91 [°C]

Power of emitter 73 [%]

Dwell time 1.7 [s]

Fixation temperature 210-213 [°C]

Power of emitter 78 [%]

Dwell time 7.2 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Black CC-R x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature 88-90 [°C]

Power of emitter 100 [%]

Dwell time 1.2 [s]

Fixation temperature 210-212 [°C]

Power of emitter 84 [%]

Dwell time 5.4 [s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature 90-93 [°C]

Power of emitter 90 [%]

Dwell time 1.2 [s]

Fixation temperature 209-211 [°C]

Power of emitter 78 [%]

Dwell time 5.4 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature 89-93 [°C]

Power of emitter 83 [%]

Dwell time 1.2 [s]

Fixation temperature 209-211 [°C]

Power of emitter 78 [%]

Dwell time 5.4 [s]

After dyeing the standard after treatment (reduction clearing) is done

Dianix® Rubine CC x [g/l] (Pale, Medium and Dark)

Standard recipe

Pale

Drying temperature	88-91	[°C]
Power of emitter	77	[%]
Dwell time	1.7	[s]

Fixation temperature	209-211	[°C]
Power of emitter	89	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done.

Medium

Drying temperature	89-93	[°C]
Power of emitter	77	[%]
Dwell time	1.7	[s]

Fixation temperature	210-212	[°C]
Power of emitter	82	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Dark

Drying temperature	89-91	[°C]
Power of emitter	72	[%]
Dwell time	1.7	[s]

Fixation temperature	210-212	[°C]
Power of emitter	77	[%]
Dwell time	7.2	[s]

After dyeing the standard after treatment (reduction clearing) is done

Color fastness to washing (ISO 105 F10)

All the dyed fabrics were tested for their color fastness to wash and color fastness to dry heat (excluding pressing) to attest to the above statement and to check if the aNIR[®] technology also yields different results.

For Color fastness Test (wash fastness) the norm used was DIN ISO 105-C06:2010 with the test conditions C2S. This part of ISO 105 specifies methods intended for determining the resistance of color of textiles of all kinds and in all forms to domestic or commercial laundering procedures for normal household articles using reference detergent [52]. The result of one multiple (M) test in this case can be approximated by the results of up to five domestic or commercial launderings at temperature not exceeding 70 °C [52]. This method is designed for the detergents and bleach systems given meaning other detergents and bleach systems may require different conditions and levels of ingredients [52]. The norm uses sodium perborate which was classified as "carcinogenic, mutagenic, or toxic for reproduction" (CMR) by the European Union. This propelled the use of Sodium percarbonate instead of sodium perborate. The concentration for the use of sodium percarbonate compared to sodium perborate is

1 [g/l] Sodium perborate = 0.67 [g/l] Sodium percarbonate [53]

Test procedure

- Dyed samples are after treated (reduction clearing)
- Put specimen and multi-fiber strip in steel container
- Dissolve 4 g/l detergent at 60°C
- 0.67 g/l Sodium percarbonate
- 50 ml solution into each stainless-steel container

- 25 steel balls into each stainless-steel container
- 5 times wash at 60°C
- Remove specimen at end of every wash, rinse twice at 40°C
- Assess the change in colour of the specimen and the staining of the adjacent fabric using a grey scale

The multi fiber strip used was the James Heal Multifiber Adjacent fabric DW (ISO 105 F10)



Figure 43: James Heal Multifiber Adjacent fabric DW (own picture)

Color fastness to dry heat (excluding pressing) ISO 105-P01:1993(E)

For Color fastness to dry heat (excluding pressing) the norm used was DIN ISO 105-P01:1993(E). This part of the ISO 105 specifies a method for determining the resistance of the color of textile of all kinds and in all forms to the action of dry heat, excluding pressing, as it is used to stabilize the size and form of textile [54]. There are three tests differing in temperature which are provided; one or more can be used, depending on the requirements and the stability of the fibers and also the method is not intended for the assessment of color change during crease-resist or dyeing process [54].

Test procedure

- Dyed samples are after treated (reduction clearing)
- Place specimen together with multi fiber strip in heating device
- Time is 30 [s] at following temperatures
 - 150°C ± 2°C
 - 180°C ± 2°C
 - 210°C ± 2°C
- Remove and leave specimen 4hrs under standard temperature atmosphere
- Assess the change in colour of the specimen and the staining of the adjacent fabric using a grey scale

For assessing the specimens using the grey scale numeric rating, 5 = best, 1 = worst
The multi fiber strip used was the James Heal Multifiber Adjacent fabric (ISO 105 F10)

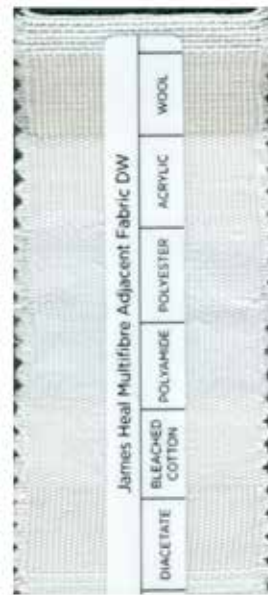


Figure 43: James Heal Multifiber Adjacent fabric DW (own picture)

4.0 Results and Discussions

This chapter explains, analyses, and compares the methods discussed before. These involve

- The first 8 samples of 6 dyed fabric samples with Dianix[®] Black CC-R and 2 blank samples without dye using convection and aNIR[®] technology
- Dyeing of all Dianix[®] XF2 disperse dyes to set standard depths using convection and aNIR[®] technology process
- Dyeing with different concentration of antimigration agents (Sera[®] Gal M-SPR) and dying with Dianix[®] Trichromic CC Brown using different drying and fixation combinations of convection and aNIR[®] technology process to analyse the migration effect. Dyeing with Dianix[®] Trichromic CC Brown recipe using Rotating Atomizer (spray) technology including different drying and fixation combinations were also made to check the migration effect
- Dyeing with aNIR[®] drying setup change technology using Dianix[®] Trichromic CC / XF2 Brown recipe of the Rotating Atomizer (spray) technology and then a final dyeing of Dianix[®] XF2/CC disperse dyes to set standard depths using convection and aNIR[®] technology process (setup 2)

4.1. Comparison of Dianix[®] Black CC-R dyes (convection process to aNIR[®] technology)

The first samples were dyed with Dianix[®] Black CC-R / Black 300% XF2 and 2 blank samples. These blank samples were dyed without dye and both samples were treated using convection and aNIR[®] technology.

Primarily, the first trial samples were made with a trial concentration of dye shades with the Dianix[®] Black CC-R. It was done to first analyze the nature of the dyes and how the machine parameters of the aNIR[®] technology process can be adjusted to get good results.

Their color strength results can be seen in Table 5. Table 5 shows that convection process yields a face and back color strength of 109 % where the face has a standard value of 100 %. aNIR[®] technology results in a lesser value of 77 % indicating more color yield on the face of the fabric than the back.

Table 6 shows the results of the color strength comparison of convection process and aNIR[®] technology with Dianix[®] Black CC-R dyed fabric. The face of both processes is compared, where aNIR[®] technology had 15 % less color strength than that of the convection process. The comparison of the back of both processes reveals a color strength of 59,76 %, meaning aNIR[®] technology yields 40,24 % less. This means dyes in the aNIR[®] technology migrate more to the face of the fabric than during the convection process. The face and back images of these samples are displayed in Figures 47,48 and 49. All other images can be seen in the Annex.

Table 5: 30 g/l Dianix[®] Black CC-R (Trial 1)

H = convection process

Standard 100%	Sample	Color Strength [%]
H 30 g/l Dianix [®] Black CC-R new face	H 30 g/l Dianix [®] Black CC-R back	109,29
aNIR [®] 30 g/l Dianix [®] Black CC-R new face	aNIR [®] 30 g/l Dianix [®] Black CC-R back	77,16

Table 6: comparison of 30 g/l Dianix[®] Black CC-R convection process and aNIR[®] technology (Trial 1)

H = convection process

Standard 100%	Sample	Color Strength [%]
H 30g/l Dianix [®] Black CC-R new face	aNIR [®] 30 g/l Dianix [®] Black CC-R new face	84,65
H 30g/l Dianix [®] Black CC-R new back	aNIR [®] 30 g/l Dianix [®] Black CC-R new back	59,76



Figure 45: Convection process 30 g/l Dianix[®] Black CC-R (face/back) (own picture)



Figure 44: aNIR[®] technology 30 g/l Dianix[®] Black CC-R (own picture)

Blank sample dyeing

After the dyeing of the samples, it was noticed that there was a migration effect. The reason being that the anti-migration agent used influenced the dyeing process which is resulting in this finding. Dyeing of the blank polyester fabric (blind dyeing) was produced with acrylate based antimigration agent; Sera[®] Print M-DP. This was done at different thermofixation temperatures of 200°C and 215°C to discover what happens to the fabric at this higher temperature. There was a change of whiteness, heat influence on the fibers (differential scanning calorimetry) compared to the normal polyester fabric, see Figure 50 to 52. The result showed slight yellowing of the polyester fabric which may be caused by the high temperature being exposed to the fabric.



Figure 49: Blank sample (without dye) Thermofixed 200°C

Figure 48: Normal PET fabric



Figure 47: Blank sample (without dye) Thermofixed 215 °C

Figure 46: Normal PET fabric

Clearly the switch from convection process to aNIR[®] technology gave an inverse value. This was an effect of various causes which another test of dyeing consistency was done.

During these trials, a 6 meter already dyed Dianix[®] Black CC-R fabric is measured by the colorimetric machine at interval steps of

- Step 1 - 0.6 m
- Step 2 - 1.2 m
- Step 3 - 1.8 m
- Step 4 - 2.4 m
- Step 5 - 3 m
- Step 6 - 3.6 m
- Step 7 - 4.2 m
- Step 8 - 4.8 m
- Step 9 - 5,4 m

The consistency of the color strength is obtained after the standard fixation temperature is reached. This is because the aNIR[®] machine needs time to run for some minutes and the parameters need to gradually be set at the set standard parameter. To reach this standard fixation temperature time becomes a major factor and that is what reflects in step 1 to step 5 (Figure 53 and 54). It was evaluated that after the standard fixation temperature is obtained, the consistency of the dyed fabric gives a $\pm 5\%$ colour strength. This indicates that the dyeing consistency of the dyed fabric is good enough and does not contribute massively to this face and back differences leading to the migration effect.

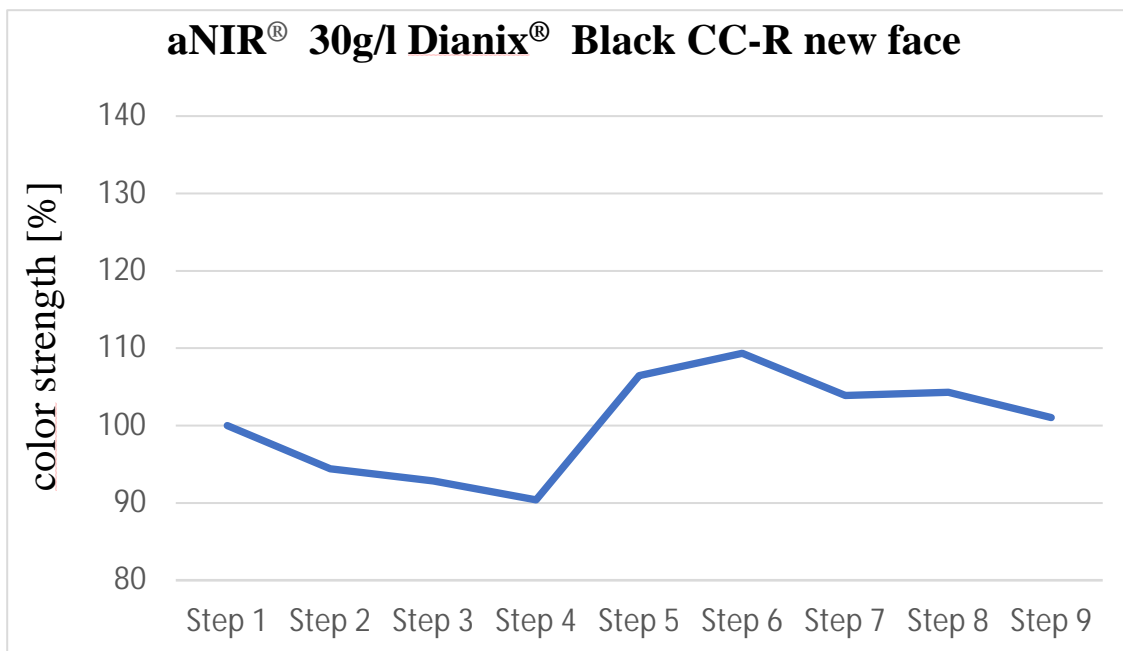


Figure 51: Graph of aNIR[®] 30g/l Dianix[®] Black CC-R new face (own graph)

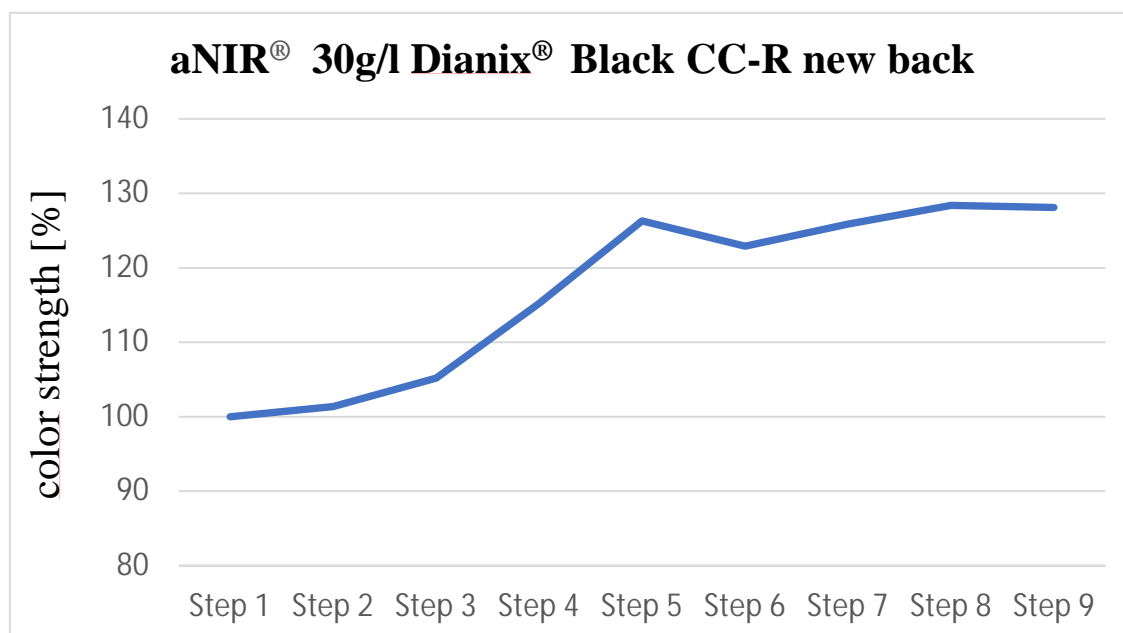


Figure 50: Graph of aNIR[®] 30g/l Dianix[®] Black CC-R new back (own graph)

4.2. Dyeing of Dianix® XF2 disperse dyes to set standard depth

Dyeing of all Dianix® XF2 disperse dyes to set standard depths using convection and aNIR® technology process.

The previous dyed samples help to establish the right adjustment of the power parameters to be set for the dyeing process. Thereafter, first trials with all Dianix® XF2 disperse dyes by both convection and aNIR® technology process are produced with the set standard recipe, standard after treatment (reduction clearing) and color fastness evaluation.

Again, all the Dianix® XF2 dispersed dyed samples resulted in higher color yield on the face of the fabric compared to the back of the fabric (migration effect) (Table 7). This is because of high evaporation rate of water molecules due to this aNIR® radiation. This radiation only excites the water molecules that is being hit on the surface. The surface water gets excited and evaporates very quickly which creates the transport effect where the liquor travels very fast to the surface, including the dyestuff.

Many factors can be contributing to this migration effect:

- distribution of dyestuff not reaching the core of the dyed fabric
- contact time of the radiation on the dyed fabrics in aNIR® technology drying and fixation phases.
- one sided illumination of either the drying or fixation phase of the aNIR® technology

Table 7: Dianix® Rubine XF2 (Pale, Medium and Dark)

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Rubine XF2 Pale face	H Dianix® Rubine XF2 Pale back	106,75
H Dianix® Rubine XF2 Medium face	H Dianix® Rubine XF2 Medium back	117,32
H Dianix® Rubine XF2 Dark face	H Dianix® Rubine XF2 Dark back	113,51
aNIR® Dianix® Rubine XF2 Pale face	aNIR® Dianix® Rubine XF2 Pale back	71,24
aNIR® Dianix® Rubine XF2 Medium face	aNIR® Dianix® Rubine XF2 Medium back	67,15
aNIR® Dianix® Rubine XF2 Dark face	aNIR® Dianix® Rubine XF2 Dark back	89,36

Scanned images of Dianix® Rubine XF2



Figure 52: aNIR® Dianix® Rubine XF2 Dark face and back (own picture)

See Annex 1 for all images shades, Annex 2,3 color fastness evaluation.

Table 8: Dianix® Yellow Brown XF2 (Pale, Medium and Dark)

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Yellow Brown XF2 Pale face	H Dianix® Yellow Brown XF2 Pale back	112,49
H Dianix® Yellow Brown XF2 Medium face	H Dianix® Yellow Brown XF2 Medium back	110,72
H Dianix® Yellow Brown XF2 Dark face	H Dianix® Yellow Brown XF2 Dark back	114,47
aNIR® Dianix® Yellow Brown XF2 Pale face	aNIR® Dianix® Yellow Brown XF2 Pale back	65,09
aNIR® Dianix® Yellow Brown XF2 Medium face	aNIR® Dianix® Yellow Brown XF2 Medium back	70,38
aNIR® Dianix® Yellow Brown XF2 Dark face	aNIR® Dianix® Yellow Brown XF2 Dark back	78,62

Table 9: Dianix® Navy XF2 [g/l] (Pale, Medium and Dark)

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Navy XF2 Pale face	H Dianix® Navy XF2 Pale back	130,15
H Dianix® Navy XF2 Medium face	H Dianix® Navy XF2 Medium back	108,34
H Dianix® Navy XF2 Dark face	H Dianix® Navy XF2 Dark back	116,62
aNIR® Dianix® Navy XF2 Pale face	aNIR® Dianix® Navy XF2 Pale back	73,75
aNIR® Dianix® Navy XF2 Medium face	aNIR® Dianix® Navy XF2 Medium back	70,16
aNIR® Dianix® Navy XF2 Dark face	aNIR® Dianix® Navy XF2 Dark back	83,32

Table 10: Dianix® Black 300% XF2 [g/l] (Pale, Medium and Dark)

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Black XF2 300% Pale face	H Dianix® Black XF2 300% Pale back	127,43
H Dianix® Black XF2 300% Medium face	H Dianix® Black XF2 300% Medium back	109,59
H Dianix® Black XF2 300% Dark face	H Dianix® Black XF2 300% Dark back	116,36
aNIR® Dianix® Black XF2 300% Pale face	aNIR® Dianix® Black XF2 300% Pale back	61,10
aNIR® Dianix® Black XF2 300% Medium face	aNIR® Dianix® Black XF2 300% Medium back	75,99
aNIR® Dianix® Black XF2 300% Dark face	aNIR® Dianix® Black XF2 300% Dark back	94,96

Additionally, all the Dianix® XF2 disperse dyed samples were tested for color fastness to washing and to dry heat.

It was evaluated that all pale shades displayed better fastness values than those of the medium and darker shades in both convection and aNIR® Dianix® disperse dyed samples. This was because of the dyestuff concentration used 1:6 standard depth in pale shades having maximum 9.5 g/l, compared to 1:1 in medium shades of minimum 15 g/l and 2:1 for dark shades having a minimum of 30 g/l. Pale shades had lower dyestuff concentrations than those of medium and dark shades.

Furthermore, it was observed that all aNIR® Dianix® XF2 disperse dyed samples had worse color fastness values to washing than those of the Dianix® XF2 convection process (see Figure 57 and 58, and Annex) This result was because most of the dyestuff were not fixed completely into the fabric compared to that of the thermosoling of the convection process. The migration effect could be a factor because of more dyestuff being migrated to one surface of the fabric.

More can be explained in the colorimetric measurement where it is clearly noticed that the color strength of the aNIR[®] Dianix[®] Rubine XF2 disperse dyed sample in Table 7, which displayed about 35 % more color strength on the face than the back of the polyester fabric. Whereas in the convection process they have about 30% more on the back than that of the face.

Similarly, the results for color fastness to dry heat of aNIR[®] Dianix[®] XF2 disperse dyed samples had worse fastness values (as seen on the multi fiber strip) than that of the Dianix[®] XF2 convection process. This is likewise because most of the dyes are not distributed completely into the core of the dyed fabric compared to that of the convection process.

Dianix[®] Rubine XF2 (All sample images are own pictures)

convection process (Dark shade)



sample after washing



original multi fiber strip



Figure 53: Color fastness to wash (convection process Dianix[®] Rubine XF2 dark shade)

aNIR[®] process (Dark shade)



sample after washing



original multi fiber strip



Figure 54: Color fastness to wash (aNIR[®] process Dianix[®] Rubine XF2 dark shade)

Medium shade



Convection process



aNIR® technology



Figure 55: Color fastness to dry heat (Convection and aNIR® process Dianix® Rubine XF2 medium shade)

4.3. Evaluation of the migration effect using trichromic recipes

Dyeing with different concentration of the antimigration agents (Sera[®] Gal M-SPR) and dying with Dianix[®] CC Trichromic Brown using different drying and fixation combination of convection and aNIR[®] technology process to analyze the migration effect. Dyeing with Dianix[®] CC Trichromic Brown recipe using Rotating Atomizer (spray) technology including different drying and fixation combination was also performed to check the cause of the migration effect.

It is evaluated that there is migration in the dyed fabric which might be caused by the antimigration agents used, drying or fixation phase of the system or any other discovery anywhere in the system. The standard antimigration agent used is Sera[®] Print M-DP. The use of different antimigration agents will help to better evaluate the effect of migration in these processes. Hence, Sera[®] Gal M-SPR was selected, involving concentrations of 7 g/l (minimum) and 30 g/l (maximum) to be dyed with Dianix[®] Black CC-R.

10 g/l Sera[®] Print M-DP is the standard concentration of antimigration agent which is used in this study. In Table 10, the value for convection process 10 g/l Sera[®] Print dyed samples yields 109,29 %. This means that the standard 10 g/l Sera[®] Print M-DP result works. With the 7 g/l Sera[®] Gal (minimum) concentration, the value shows a difference of 0.58 % indicating that, these antimigration concentrations also produce good results in the convection process. After increasing the antimigration agent concentration to 30 g/l Sera[®] Gal (maximum) in the convection process, the value gets worse with a value of 125,93 %. This signifies that migration increases when more antimigration agent is used and at the moment there is no definitive conclusion for this behaviour.

In the aNIR[®] 30 g/l Dianix[®] Black CC-R 10 g/l Sera[®] Print M-DP face gets worse with a value of 77,16 %. In the aNIR[®] 30g/l Dianix[®] Black CC-R 7 g/l Sera[®] Gal M-SPR, the color strength still gets worse having a value of 64,43 %. Adding more antimigration agents produce worse results.

The result concluded that the use of the standard antimigration of 10 g/l Sera[®] Print for all further sample can produces good results. Also, the migration effect of different face back values still existed which now prompted the use of different drying and thermofixation combination in the processes.

Table 11: 30 g/l Dianix® Black CC-R 7/30 [g/l] (minimum and maximum concentration)

H = convection process

Standard 100%	Sample	Color Strength [%]
H 30 g/l Dianix® Black CC-R 10 g/l Sera® Print M-DP face	H 30 g/l Dianix® Black CC-R 10 g/l Sera® Print M-DP back	109,29
H 30 g/l Dianix® Black CC-R 7 g/l Sera® Gal M-SPR face	H 30 g/l Dianix® Black CC-R 7 g/l Sera® Gal M-SPR back	108,71
H 30 g/l Dianix® Black CC-R 30 g/l Sera® Gal M-SPR face	H 30 g/l Dianix® Black CC-R 30 g/l Sera® Gal M-SPR back	125,93
aNIR® 30 g/l Dianix® Black CC-R 10 g/l Sera® Print M-DP face	aNIR® 30 g/l Dianix® Black CC-R 10 g/l Sera® Print M-DP back	77,16
aNIR® 30g/l Dianix® Black CC-R 7 g/l Sera® Gal M-SPR face	aNIR® 30 g/l Dianix® Black CC-R 7 g/l Sera® Gal M-SPR back	64,43
aNIR® 30 g/l Dianix® Black CC-R 30 g/l Sera® Gal M-SPR face	aNIR® 30 g/l CC-R new Dianix® Black CC-R 30 g/l Sera® Gal M-SPR back	62,48



Figure 56: Convection process 30 g/l Dianix® Black CC-R 7 g/l Sera® Gal M-SPR (own picture)

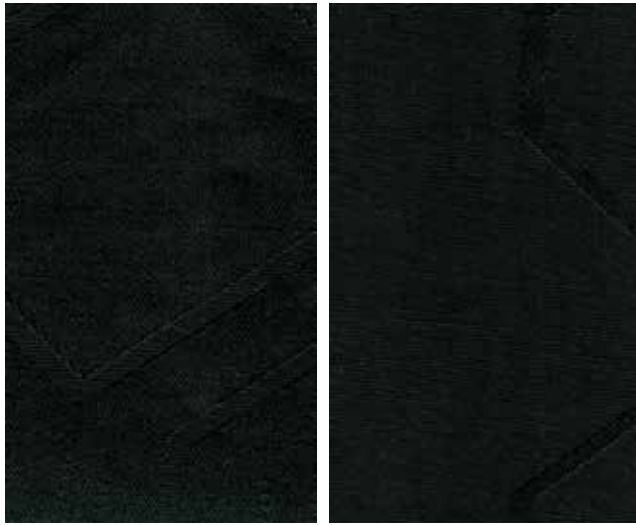


Figure 58: Convection process 30 g/l Dianix[®] Black CC-R 30 g/l Sera[®] Gal M-SPR (own picture)



Figure 57: aNIR[®] 30 g/l Dianix[®] Black CC-R 7 g/l Sera[®] Gal M-SPR (own picture)

Dianix® Trichrome CC Brown combinations

As said earlier, different combinations of drying and fixation with Dianix® CC Trichromic Brown are produced to analyze where the migration effect is present. The ambient air dried with convection thermosoling had a face/back value of 119,6 %. This explains that a change in the drying and thermosoling phase using the convection process produces good results with less migration. Using aNIR® thermofixation with ambient air dried gets worse with the back producing 30 % more color strength. It gets worse when aNIR® technology is used in both drying and fixation. This explains that during the drying in the aNIR® technology, the dyes migrate towards the surface of the fabric. After which the dyes are fixed where they migrate to, which in this case is the face of the fabric yielding 58 % color strength. All these results can be seen in Table 12.



Figure 59 : aNIR® 30 g/l Dianix® Black CC-R 30 g/l Sera® Gal M-SPR (own picture)

Table 12: Dianix® Trichrome CC Brown combinations

Standard 100%	Sample	Color Strength (%)
Ambient air dried and convection oven thermosoling face	Ambient air dried and convection oven thermosoling back	119,6
Ambient air dried and aNIR® thermofixed face	Ambient air dried and aNIR® thermofixed back	130,28
aNIR® dried and aNIR® thermofixed face	aNIR® dried and aNIR® thermofixed back	58,21

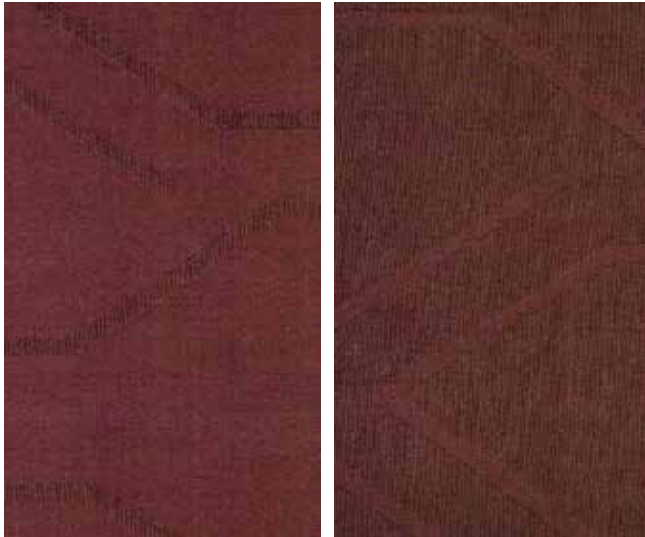


Figure 62: Dianix[®] Trichrome CC Brown ambient air dried with convection oven thermosoling (own picture)

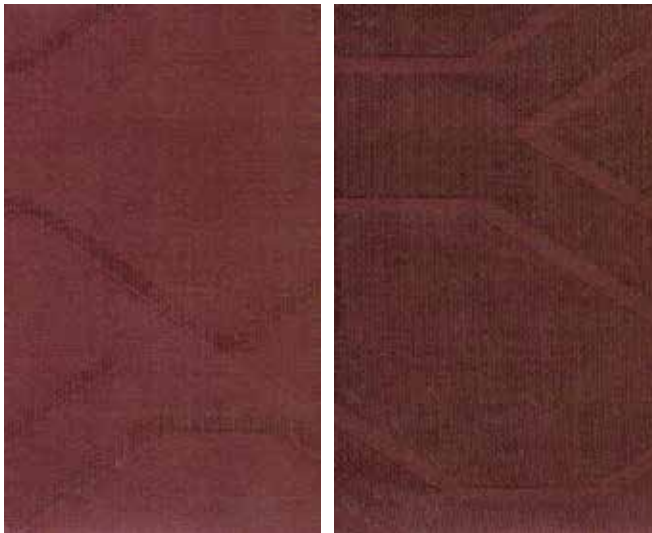


Figure 60: Dianix[®] Trichrome CC Brown ambient air dried with aNIR[®] thermofixation (own picture)

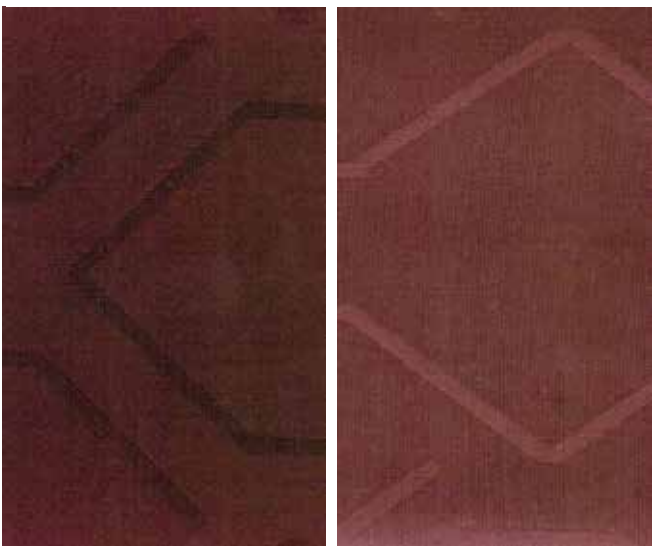


Figure 61: Dianix[®] Trichrome CC Brown aNIR[®] technology process (own picture)

Rotating Atomizer (spray) technology Dianix® Trichrome CC Brown

After producing the earlier process, other trial samples are done also with the same Dianix® Trichromic CC and XF2 brown to compare the padding (conventional process) with minimum application (Rotating Atomizer (spray)). Similarly, with the same drying and thermofixation combination processes, Dianix® Trichromic CC Brown shows that the Rotating Atomizer (spray) application dried with ambient air and convection oven thermosoling have good results with 96,44 % colour strength. The aNIR® thermofixation process produces worse results when the rotating atomizer dyed fabric is dried with ambient air. This indicates that there is still dyestuff migrating after the dye is minimally applied in combination with aNIR® thermofixation. The aNIR® drying and aNIR® thermofixation value also indicates that there is migration present but the value of 113 % is unusual, considering that the aNIR® process normally produces more color strength on the face side of the fabric. This value might be an outlier in the colometric measurement or be caused during the transportation of the dyed fabric from WECO to Hof University of Applied Sciences, Münchberg.

Table 13: Rotating Atomizer (spray) technology Dianix® Trichrome CC Brown

Standard 100%	Sample	Color Strength (%)
Rotating Atomizer (spray) Ambient air dried and convection oven thermosoling face	Rotating Atomizer (spray) Ambient air dried and convection oven thermosoling back	96,44
Rotating Atomizer (spray) Ambient dried and aNIR® thermofixed face	Rotating Atomizer (spray) Ambient dried and aNIR® thermofixed back	124,71
Rotating Atomizer (spray) aNIR® dried and aNIR® fixed face	Rotating Atomizer (spray) aNIR® dried and aNIR® fixed back	113,05



Figure 65: Rotating Atomizer (spray) technology Dianix[®] Trichrome CC Brown (Ambient air dried with convection oven thermosoling) (own picture)



Figure 63: Rotating Atomizer (spray) technology Dianix[®] Trichrome CC Brown (Ambient air dried with aNIR[®] fixation) (own picture)

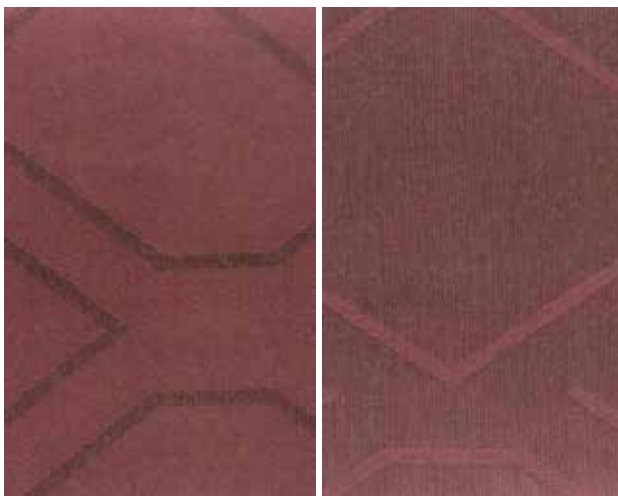


Figure 64: Rotating Atomizer (spray) technology Dianix[®] Trichrome CC Brown (aNIR[®] technology process) (own picture)

With the use of Dianix® Trichromic XF2 Brown, the values get similarly worse compared to all the drying and fixation combinations. Rotating Atomizer (spray) ambient air-dried and convection oven thermosoling show a value of 138 %. This is because migration of the dyes happens during the drying or fixation process. Similarly, ambient air-dried sample thermofixed with aNIR® yield 134,93%. The results indicate that when the drying phase is changed to ambient air, the values for both samples are similar. It gets worse when aNIR® drying and thermofixation is produced explaining that the migration effect happens in the drying phase. Table 14 reveals these results.

Table 14: Rotating Atomizer (spray) technology Dianix® Trichrome XF2 Brown

Standard 100%	Sample	Color Strength (%)
Rotating Atomizer (spray) ambient dried and convection oven thermosoling face	Rotating Atomizer (spray) ambient dried and convection oven thermosoling back	138,93
Rotating Atomizer (spray) ambient dried and aNIR® thermofixed face	Rotating Atomizer (spray) ambient dried and aNIR® thermofixed back	134,93
Rotating Atomizer (spray) aNIR® dried and aNIR® thermofixed face	Rotating Atomizer (spray) aNIR® dried and aNIR® thermofixed back	143,35

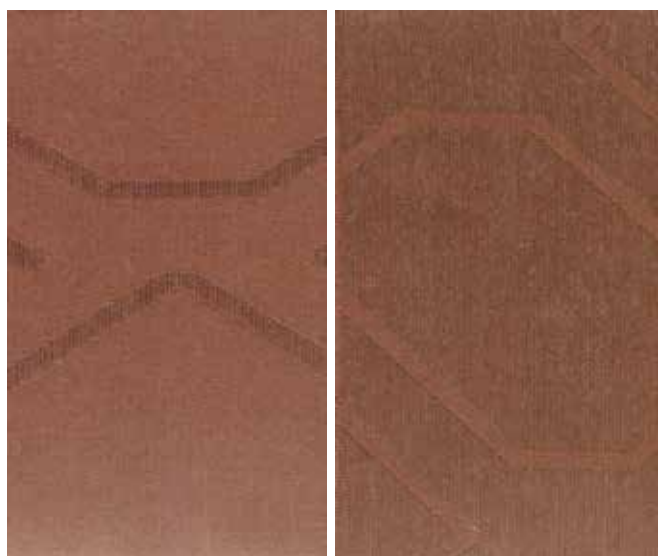


Figure 66: Rotating Atomizer (spray) technology Dianix® Trichrome XF2 Brown (Ambient air dried with convection oven thermosoling) (own picture)

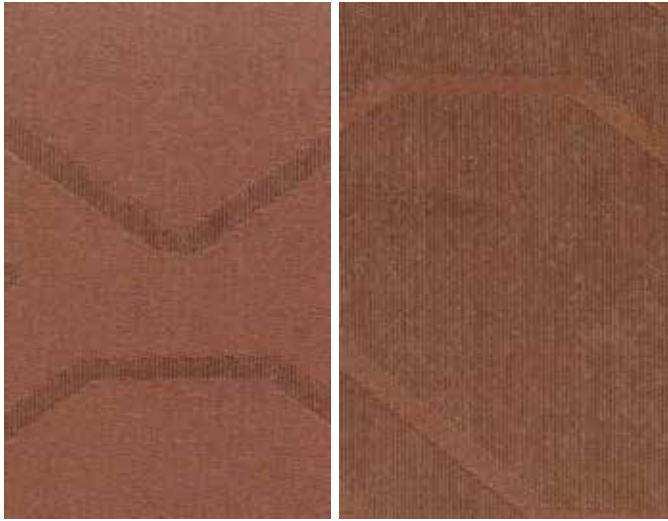


Figure 67: Rotating Atomizer (spray) technology Dianix[®] Trichrome XF2 Brown (Ambient air dried with aNIR[®] fixation) (own picture)

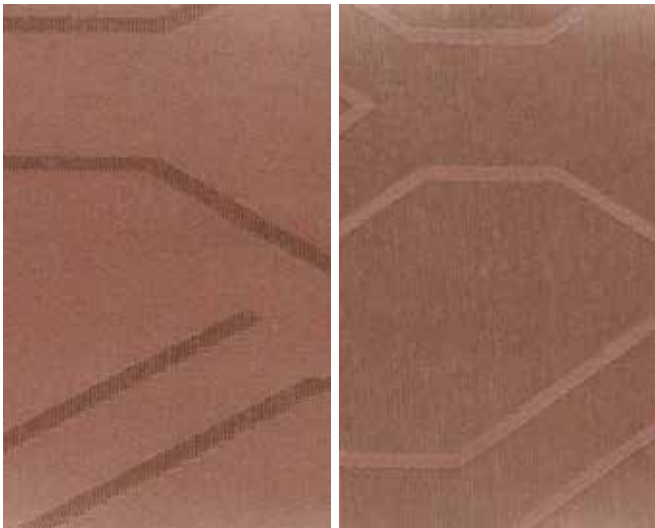


Figure 68: Rotating Atomizer (spray) technology combination Dianix[®] Trichrome XF2 Brown (aNIR[®] technology process) (own picture)

4.4. Final dyeing using updated process configuration

Dyeing with aNIR[®] drying setup change technology using Dianix[®] Trichrome CC / XF2 Brown recipe of the Rotating Atomizer (spray) technology and then a final dyeing of Dianix[®] XF2/CC disperse dyes according to set standard depths using convection and aNIR[®] technology process (setup 2)

The results for the previous test shows that the drying phase of the aNIR[®] technology played an influential role in the migration effect. This led to a setup change in the drying phase and the aNIR[®] technology with Dianix[®] Trichrome CC / XF2 Brown recipe and with the Rotating Atomizer (spray) technology samples also repeated with results shown in Table 15 to Table 18. This second setup result shows an opposite effect which makes all the sample test better. The Dianix[®] Trichrome CC Brown aNIR[®] drying and aNIR[®] thermofixed samples show 136 % colour strength meaning that, the dye in the fabric now migrates in both direction of the drying lamp emitters but with an inverse result compared to that of setup 1. All other dyes and combinations also yield similar results but mainly giving the inverse results between setup 1 and setup 2.

Table 15: Dianix[®] Trichrome CC Brown aNIR[®] technology Setup 2

Standard 100%	Sample	Color Strength (%)
aNIR [®] drying and aNIR [®] thermofixed face	aNIR [®] drying and aNIR [®] thermofixed back	136,84

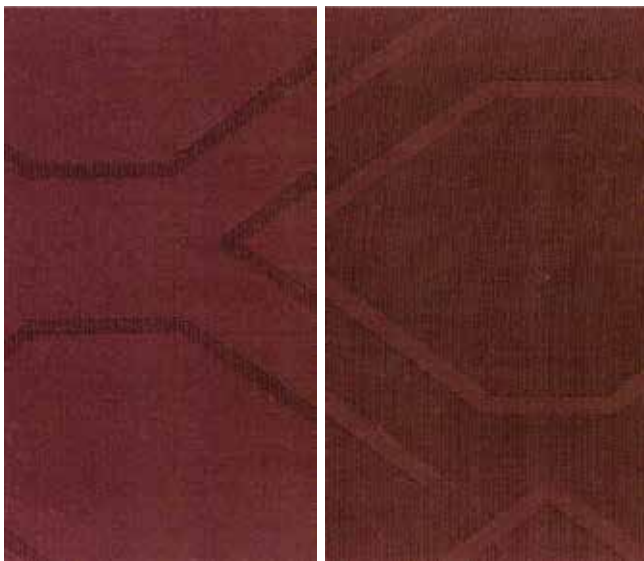


Figure 69: Dianix[®] Trichrome CC Brown aNIR[®] technology Setup 2 (face/back) (own picture)

Table 16: Dianix® Trichrome XF2 Brown aNIR® technology Setup 2

Standard 100%	Sample	Color Strength (%)
aNIR® drying and aNIR® thermofixed face	aNIR® drying and aNIR® thermofixed back	145,95

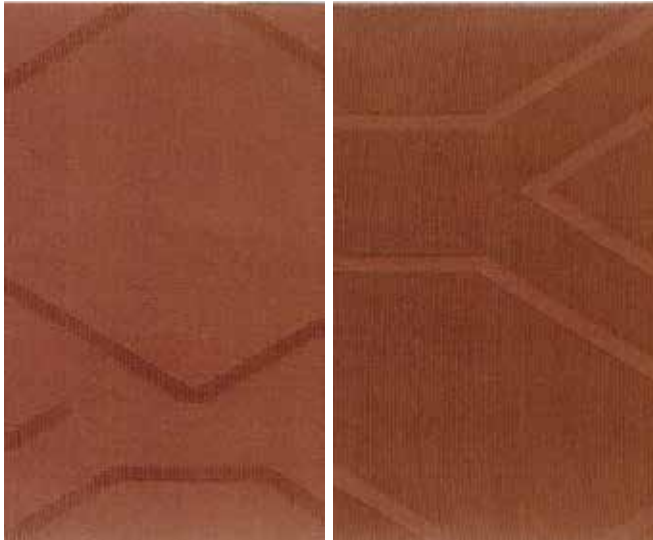


Figure 70: Dianix® Trichrome XF2 Brown aNIR® technology Setup 2(face/back) (own picture)

Table 17: Rotating Atomizer (spray) technology with aNIR® technology process (Dianix® Trichrome CC Brown) setup 2

Standard 100%	Sample	Color Strength (%)
aNIR® drying and aNIR® thermofixed face	aNIR® drying and aNIR® thermofixed back	171,30



Figure 71: Rotating Atomizer (spray) technology with aNIR® technology process (Dianix® Trichrome CC Brown) setup 2 (own picture)

Table 18: Rotating Atomizer (spray) technology with aNIR® technology process Dianix® Trichrome XF2 Brown) setup 2

Standard 100%	Sample	Color Strength (%)
aNIR® drying and aNIR® thermofixed face	aNIR® drying and aNIR® thermofixed back	175,14

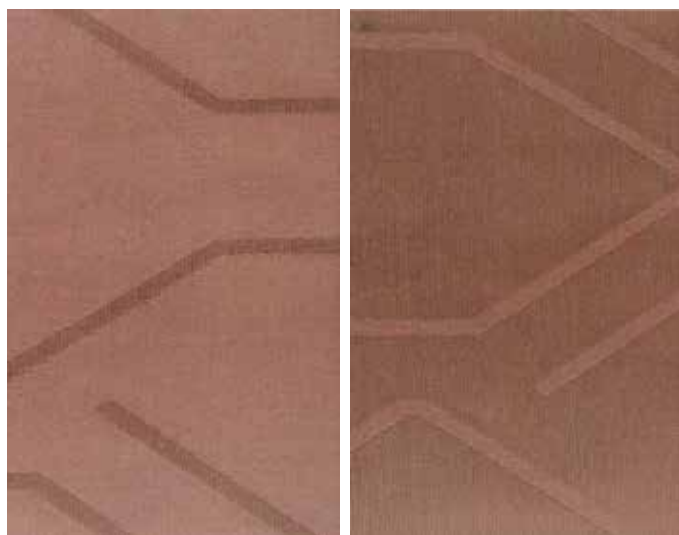


Figure 72: Rotating Atomizer (spray) technology with aNIR® technology process (own picture)

Comparing fixation yield of convection and aNIR® Dianix® CC Trichromic brown between Setup 1 and Setup 2

Table 19 shows the comparison between the fixation yield of setup 1 and setup 2 with convection and aNIR® technology using the Dianix® CC Trichromic brown. When the aNIR® technology face of the setup 1 is compared to convection process the value show 134,0 % which that aNIR® technology face has more dyes migrating to the surface of the fabric making it worse. In the setup 2, the aNIR® technology face compared to convection process face show has a value of 94,81 %. This explains that aNIR® technology setup 2 face produces good results almost similar to that of the convection process. Alongside with the back of these setups, the results for setup 1 still gets worse with a value of 75 % and setup 2 yields inverse results which is 125 %.

Table 19: Comparison fixation yield of convection and aNIR® Dianix® CC Trichromic brown (Setup 1 and Setup 2)

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength (%)
H Drying and fixation face	(Setup 1) aNIR® drying and aNIR® thermofixed face	134,0
H Drying and fixation face	(Setup 2) aNIR® drying and aNIR® thermofixed face	94,81
H Drying and fixation back	(Setup 1) aNIR® drying and aNIR® thermofixed back	75,0
H Drying and fixation back	(Setup 2) aNIR® drying and aNIR® thermofixed back	125,0

Table 20: Dianix® Rubine XF2 (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Rubine XF2 Pale face	H Dianix® Rubine XF2 Pale back	106,75
H Dianix® Rubine XF2 Medium face	H Dianix® Rubine XF2 Medium back	117,32
H Dianix® Rubine XF2 Dark face	H Dianix® Rubine XF2 Dark back	113,51
aNIR® Dianix® Rubine XF2 Pale face	aNIR® Dianix® Rubine XF2 Pale back	146,54
aNIR® Dianix® Rubine XF2 Medium face	aNIR® Dianix® Rubine XF2 Medium back	141,74
aNIR® Dianix® Rubine XF2 Dark face	aNIR® Dianix® Rubine XF2 Dark back	142,63

aNIR® Dianix® Rubine XF2

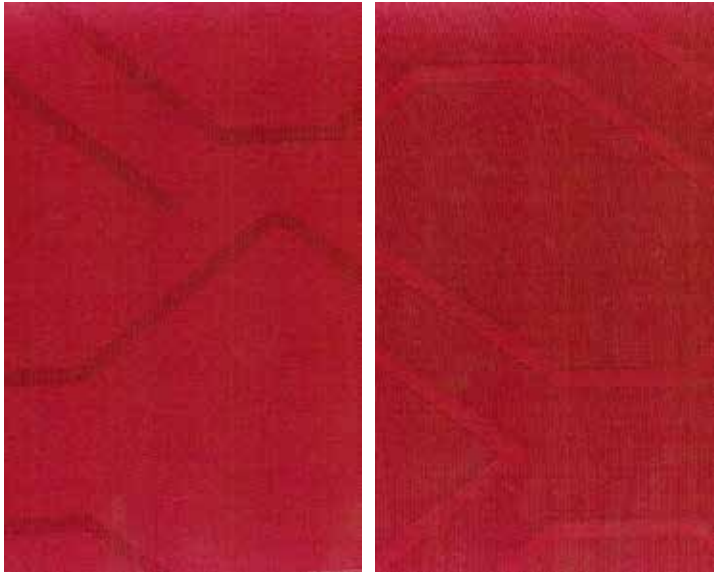


Figure 73: aNIR® Dianix® Rubine XF2 setup 2 dark shade (own picture)

Table 21: Dianix® Yellow Brown XF2 (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Yellow Brown XF2 Pale face	H Dianix® Yellow Brown XF2 Pale back	112,49
H Dianix® Yellow Brown XF2 Medium face	H Dianix® Yellow Brown XF2 Medium back	110,72
H Dianix® Yellow Brown XF2 Dark face	H Dianix® Yellow Brown XF2 Dark back	114,47
aNIR® Dianix® Yellow Brown XF2 Pale face	aNIR® Dianix® Yellow Brown XF2 Pale back	151,46
aNIR® Dianix® Yellow Brown XF2 Medium face	aNIR® Dianix® Yellow Brown XF2 Medium back	137,18
aNIR® Dianix® Yellow Brown XF2 Dark face	aNIR® Dianix® Yellow Brown XF2 Dark back	125,09

Table 22: Dianix® Navy XF2 (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Navy XF2 Pale face	H Dianix® Navy XF2 Pale back	130,15
H Dianix® Navy XF2 Medium face	H Dianix® Navy XF2 Medium back	108,34
H Dianix® Navy XF2 Dark face	H Dianix® Navy XF2 Dark back	116,62
aNIR® Dianix® Navy XF2 Pale face	aNIR® Dianix® Navy XF2 Pale back	143,16
aNIR® Dianix® Navy XF2 Medium face	aNIR® Dianix® Navy XF2 Medium back	135,16
aNIR® Dianix® Navy XF2 Dark face	aNIR® Dianix® Navy XF2 Dark back	127,55

Table 23: Dianix® Black 300% XF2 (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Black XF2 300% Pale face	H Dianix® Black XF2 300% Pale back	127,43
H Dianix® Black XF2 300% Medium face	H Dianix® Black XF2 300% Medium back	109,59
H Dianix® Black XF2 300% Dark face	H Dianix® Black XF2 300% Dark back	116,36
aNIR® Dianix® Black XF2 300% Pale face	aNIR® Dianix® Black XF2 300% Pale back	135,96
aNIR® Dianix® Black XF2 300% Medium face	aNIR® Dianix® Black XF2 300% Medium back	120,36
aNIR® Dianix® Black XF2 300% Dark face	aNIR® Dianix® Black XF2 300% Dark back	109,76

Table 24: aNIR® Dianix® Rubine CC (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Rubine CC Pale face	H Dianix® Rubine CC Pale back	128,43
H Dianix® Rubine CC Medium face	H Dianix® Rubine CC Medium back	104,92
H Dianix® Rubine CC Dark face	H Dianix® Rubine CC Dark back	117,12
aNIR Dianix® Rubine CC Pale face	aNIR Dianix® Rubine CC Pale back	137,95
aNIR Dianix® Rubine CC Medium face	aNIR Dianix® Rubine CC Medium back	120,74
aNIR Dianix® Rubine CC Dark face	aNIR Dianix® Rubine CC Dark back	117,06

Table 25: aNIR® Dianix® Yellow Brown CC (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Yellow Brown CC Pale face	H Dianix® Yellow Brown CC Pale back	105,25
H Dianix® Yellow Brown CC Medium face	H Dianix® Yellow Brown CC Medium back	114,27
H Dianix® Yellow Brown CC Dark face	H Dianix® Yellow Brown CC Dark back	122,54
aNIR® Dianix® Yellow Brown CC Pale face	aNIR® Dianix® Yellow Brown CC Pale back	150,55
aNIR® Dianix® Yellow Brown CC Medium face	aNIR® Dianix® Yellow Brown CC Medium back	138,90
aNIR® Dianix® Yellow Brown CC Dark face	aNIR® Dianix® Yellow Brown CC Dark back	120,35

Table 26: aNIR® Dianix® Navy CC (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Navy CC Pale face	H Dianix® Navy CC Pale back	116,82
H Dianix® Navy CC Medium face	H Dianix® Navy CC Medium back	108,53
H Dianix® Navy CC Dark face	H Dianix® Navy CC Dark back	112,70
aNIR® Dianix® Navy CC Pale face	aNIR® Dianix® Navy CC Pale back	141,69
aNIR® Dianix® Navy CC Medium face	aNIR® Dianix® Navy CC Medium back	143,98
aNIR® Dianix® Navy CC Dark face	aNIR® Dianix® Navy CC Dark back	124,78

Table 27: aNIR® Dianix® Black CC-R (Pale, Medium and Dark) Setup 2

Standard recipe

H = convection process

Standard 100%	Sample	Color Strength [%]
H Dianix® Black CC-R Pale face	H Dianix® Black CC-R Pale back	123,79
H Dianix® Black CC-R Medium face	H Dianix® Black CC-R Medium back	110,97
H Dianix® Black CC-R Dark face	H Dianix® Black CC-R Dark back	115,76
aNIR® Dianix® Black CC-R Pale face	aNIR® Dianix® Black CC-R Pale back	141,55
aNIR® Dianix® Black CC-R Medium face	aNIR® Dianix® Black CC-R Medium back	129,59
aNIR® Dianix® Black CC-R Dark face	aNIR® Dianix® Black CC-R Dark back	103,70

See Annex 1,2,3 for all results.

5.0 Summary

This thesis investigates how aNIR[®] radiation and its technology can be used for the continuous disperse dyeing of polyester fibers. There were two disperse dyes used, which are Dystar[®] Groups high energy dyes, Dianix[®] XF2, and their medium energy dyes, Dianix[®] CC.

These dyes revealed different dyeing characteristics which led to good results. Different experimental setups led to the identification of specific parameters, and temperatures in the aNIR[®] radiation technology.

All samples were measured revealing their color strength, comparing the face and back of the fabric with a colorimetric measurement, which provides a percentage value. All of the dyed samples showed more color strength differences between the front and back sides due to migration effects after using the aNIR[®] drying and thermofixation process (1st setup).

Therefore, experiments with different drying methods and their combinations revealed that the reproducible migration takes place strongly during the drying phase of the process.

It was evaluated that a ± 5 % consistency of the color yield in the dyed fabrics indicated a good result. Dianix[®] XF2 dyed samples using aNIR[®] technology compared to convection process generated better results than those of Dianix[®] CC dyed samples. This was due to Dianix[®] CC dyed samples demonstrating a slight tendency of more staining after color fastness to washing test (ISO 105 F10) and color fastness to dry heat (ISO 105-P01:1993(E)), after the use of the aNIR[®] technology. But in all, color fastness to washing and to dry heat of the Dianix[®] disperse dyed fabrics produced good results.

The padding application had a pick-up of around 68% while the minimal application (rotating atomizer technology) had less than half of the water content with a pick-up of 32%.

This decrease in the water content requires roughly 22% less energy input for the drying phase in the aNIR[®] technology but both application methods produce comparable results in color yield on the face side of the fabric.

In the drying phase of the aNIR[®] technology, the Dianix[®] XF2 in pale shade needed roughly 18% more energy than that of the dark shade of the same dye. Also, during the thermofixation phase of aNIR[®] technology, the same dyestuff shades show the same 18% difference in energy required. This shows that absorption depends on dyestuff concentration to reach the set process parameter (surface temperature).

In the convection process, the dyed fabric was dried in 300 seconds and thermofixed in 60 seconds. In the aNIR[®] technology (1st setup) dyed fabrics were dried in 2,5 seconds and thermofixed in 5,4 seconds. aNIR[®] technology (1st setup) showed good results but had a problem with migration effects on the dyed fabrics.

In the aNIR[®] technology (2nd setup) the drying phase reduces from 2,5 to 1,2 seconds and thermofixation occurs in 5,4 seconds. These results are comparable to those of the convection process in color yield and consistency but exceed them drastically in energy efficiency. This leads to the conclusion that using the aNIR[®] technology for the drying phase of the laboratory setup is possible, but it is necessary to use double sided illumination to get good results. For the fixation phase of the laboratory setup, one sided illumination is sufficient.

Further research must be conducted to analyze the separate process steps in more detail, but the conclusion of this thesis is that the aNIR[®] technology is suitable for the continuous disperse dyeing of polyester.

6.0. Reference

- [1] J. Koh, "Dyeing with Disperse Dyes," in *Textile Dyeing*, South Korea, www.intechopen.com, 2011, pp. 196 - 200.
- [2] H. Prof. Dr. rer. nat. Rouette, *Encyclopedia of Textile Finishing*, Aachen: Springer, 2000.
- [3] T. Bechtold and T. Pham, *Textile Chemistry*, Berlin/Boston: Walter de Gruyter GmbH, Berlin/Boston, 2019.
- [4] H. Köpnick, M. Schmidt, W. Brüggling, J. Rüter and W. Kaminsky, "Polyesters," Wiley-VCH Verlag GmbH & Co. KGaA., Weinheim, June, 2000.
- [5] FIBER-LINE® LLC, "FIBER-LINE," FIBER-LINE® LLC, [Online]. Available: <https://www.fiber-line.com/uploads/pdf%20US/fl.us.datasheet-petpolyester.pdf>.
- [6] International Rayon and Synthetic Fibres Committee (ed.), "Information on Man-Made Fibres," vol. 22, 1985.
- [7] H. Sattler and M. Schweizer, "Fibers, 5. Polyester Fibers," in *Ullmann's encyclopedia of Industrial chemistry*, Weinheim, Wiley-VCH Verlag GmbH & Co. KGaA, 2011.
- [8] M. A. Ravindranath K, "Polyethylene terephthalate - Chem., thermodynamics and transp. prop.," *Chem Eng Sci*, no. 41, 1986.
- [9] K. T. Dimzon I.K. D., "Comprehensive Analytical Chemistry," vol. 58, no. ISSN 0166-526X, 2012.
- [10] A. S., *Introduction to Plastics Engineering*, William Andrew Publishing, 2018.
- [11] H. Ludewig, *Polyesterfasern*, vol. 2nd ed., Berlin: Akademie-Verlag, 1975.
- [12] A. Ziabicki, *Fundamentals of Fibre Formation*, London: Wiley-Inter- science, 1976.
- [13] I. Hamana, M. Matsui and S. Kato, "Filament formation in Melt spinning," *Melliand Textilber*, no. 4, pp. 382 - 388, 499 - 503, 1969.
- [14] L. E, "Special Structure in Oriented Fibers Made Out of Polyethylene Terephthalate (PET)," *Kolloid ZZ Polym.*, no. 251, pp. 1028 - 1037, 1973.
- [15] H. Heuvel and R. Huisman, "Effect of winding speed on the physical structure of as-spun poly (ethylene terephthalate) fibers, including orientation-induced crystallization," *J of App. Sci.*, no. 22, pp. 2229 - 2243, 1978.
- [16] A. Ziabicki and H. Kawai, *High-Speed Fiber Spinning*, New York: Wiley-Interscience, 1985.
- [17] M. Jambrich, A. Diacikova, O. Durcova and A. Chvala, "Acta Polymer," vol. 37, pp. 118 - 121, 1986.
- [18] A. E. J. T. G.W. Davis, "Fiber Production," no. 12, pp. no. 2, 22 - 28, 1984.
- [19] G. R. H. R. W. Dietrich, *Chemiefasern/Text.Ind.*, vol. 32/84, pp. 612 - 625, 1982.
- [20] W. S. H. Lückert, *Chemiefasern/Text.Ind.*, no. 36/88, pp. 24 - 29, 1986.
- [21] A. T. I. Marshall, *J. Appl. Chem.*, vol. 4, pp. 145 - 153, 1954.
- [22] H. Berg, *Chemiefasern/Text.-Ind*, no. 22/74, pp. 215 - 222, 1972.
- [23] K. Scharley, "Bühler Nachr.," Bühler-Miag., Braunschweig, 1983.
- [24] K. Riggert, *Chemiefasern*, vol. 19, pp. 816 - 823, 1969.
- [25] M. Clark, "Dyeing of synthetic fibers," in *Handbook of textile and industrial dyeing*, Cambridge, Woodhead Pub. Ltd., 2011, pp. 46 - 70.
- [26] P. J. T. Morris, *The American Synthetic Rubber Research Program*, University of Pennsylvania Press, 1989.

- [27] P. Gulrajani, "<https://www.youtube.com/channel/UChSq2eYfRVdWqLbiWUkgx5w>," Department of Textile Processing GPHisar, 3 July 2019. [Online]. Available: <https://www.youtube.com/watch?v=NQRxwHTHKmo>. [Accessed August 2022].
- [28] H. W. H. Flath, "Textile, 4. Dyeing of Man-Made Fibers," in *Ullmann's Ency. of Ind. Chem.*, Weinheim, Wiley-VCH Verlag GmbH & Co., 2016, p. 3.
- [29] A. D. Broadbent, *Basic Principles of Textile Coloration*, Bradford, UK: SDC, ISBN 0-901956-76-7, 2001.
- [30] P. & G. P. Gordon, *Organic Chemistry in Colour*, New York: Springer-Verlag, 1983.
- [31] N. D.M.(ED), *The Dyeing of Synthetic-polymer and Acetate Fibers*, Bradford, UK: SDC, 1979.
- [32] M. I. Kiron, "Textile Learner," 6 March 2021. [Online]. Available: <https://textilelearner.net/disperse-dyes-dyeing-and-printing-method/>. [Accessed 2 July 2022].
- [33] J. F. Dawson, "Fifty Years of Disperse Dyes," *Rev. Prog. Coloration*, Vols. 14, No. 1, no. 0557-9325, p. 90–97, 1984.
- [34] T. M. Baldwinson, "Post-stentered wash fastness of disperse dyes on polyester-the significance of various test conditions," *J. Soc. Dyers Colour*, Vols. 105, No. 7-8, no. 0037-9859, p. 269–276, 1989.
- [35] J. S. & K. J. P. Koh, "Application of Phthalimide Based Alkali-clearable Azo Disperse Dyes onto Polyester and polyester/cotton Blends," *J. Soc. Dyers Colour*, vol. 114 No.4, no. 0037-9859, p. 121–12, 1998.
- [36] J. & G. A. J. Koh, "Synthesis and Application of an Alkali-clearable Azo Disperse Dye Containing a Fluorosulfonyl Group and Analysis of its Alkali- hydrolysis Kinetics," *Dyes Pigment*, Vols. 50, No. 2, no. 0143-7208, p. 117–126, 2001.
- [37] P. & G. P. Gordon, *Organic Chemistry in Colour*, New York: Springer-Verlag, 1983.
- [38] D. M. (. Nunn, *The Dyeing of Synthetic Polymers and Acetate*, Bradford: SDC, ISBN 0-901956-36-8, 1979.
- [39] H. Braun, "Particle Size and Solubility of Disperse Dyes," *Rev. Prog. Color. Relat. Top*, vol. 13, p. 62–72, 1983.
- [40] F. Jones, *J. Soc. Dyers Colour*, vol. 100, pp. no. 2, 66–72, 1984.
- [41] E. Waters, "The Dyeing of Terylene Polyester Fibre," *J. Soc. Dyers Colour*, vol. 66. No. 12, no. ISSN 0037-9859, p. 609–615, 1950.
- [42] W. Ingamells, *Colour for Textiles: A User's Handbook*, Bradford, UK: SDC, ISBN 0-901956-56-2, 1993.
- [43] T. L. & T. J. C. Dawson, "Dye Diffusion-The Key to Efficient Coloration," *J. Soc. Dyers Colour*, Vols. 95, No. 12, no. 0037-9859, p. 417–42, 1979.
- [44] H. L. (RETIRED, "Textile Dyeing, 5. Dyeing of Fiber Blends,," in *Ullmann's Ency. of Ind. Chem.*, Weinheim, Wiley-VCH Verlag GmbH & Co. KGaA, 2016, pp. 45, pp. 191–202], [55, pp. 141–142.
- [45] A. D. Broadbent, "Basic Principles of Textile Coloration," *SDC*, no. ISBN 0-901956-76-7, 2001.
- [46] H. Leube and W. Rüttiger, "Textile Dyeing, 1. General," in *Ullmanns Encyclopedia of industrial chemistry*, Weinheim, Wiley-VCH Verlag GmbH & Co. KGaA, 2016, pp. 26-29.
- [47] naba83, "wiseled," wiseled, 14 april 2021. [Online]. Available: <https://www.wiseled.com/post/what-s-the-deal-with-infrared>. [Accessed 15 September 2022].

- [48] E. V. T. D. M. Haaland, *Partial Least-Squares Methods for Spectral Analyses .1. Relation to Other Quantitative Calibration Methods and the Extraction of Qualitative Information*, American Chemical Society, 1988.
- [49] V. Nitsche, *Introduction to adphosNIR® – Drying Systems*, adphos, 2018.
- [50] C. Sivaramakrishnan, "Washing off agents and their techniques," [researchgate.net/publication/296375714](https://www.researchgate.net/publication/296375714), 2006.
- [51] DyStar® Singapore Pte Ltd., *Dianix® shadecard, Sustainable performing disperse dyes dyes to rely on*, Singapore: DyStar Singapore Pte Ltd., 2020.
- [52] The International Organization for Standardization, *ISO 105-C06; Colour fastness to domestic and commercial laundering*, Geneva: ISO copyright office, 2010.
- [53] G. F. Committee. [Online]. Available: https://www.farbechtheit.info/pdfs/studie_Na-Percarbonat-vs-Na-Perborat.pdf. [Accessed 15 September 2022].
- [54] The International Organization for Standardization, *ISO 105-P01; Colour fastness to dry heat (excluding pressing)*, Geneva: International Organization for Standardization, 1993.
- [55] WECO GMBH / RotaSpray GmbH, WECO GMBH /RotaSpray GmbH, [Online]. Available: https://www.weko.net/files/weko/downloads/Pressedownloads/RotaSpray_2021.pdf. [Accessed August 2022].
- [56] D. K. K. O. Bär, *Enhances of aNIR-Drying in Textile Printing Applications*, adphos Digital Printing GmbH, 2018.
- [57] D. D. Bär, *New developments in multi layer coating for conductive textiles with interim drying via nir technology*, Dresden: Adphos digital printing GmbH, 2021.
- [58] J. Dumbleton, *J. Polym.Sci.*, , vol. Part A 2, pp. 795 - 800, 1968.
- [59]
- [60] S. C. Liew, *Electromagnetic Waves*, Centre for Remote Imaging, Sensing and Processing, (2001)Retrieved 2006-10-27..
- [61] BASF, "Dyeing Auxiliaries," BASF, 1987. [Online]. Available: http://nicher1987.com/jbp/BASF/Dyeing/dyeing_auxiliaries.htm. [Accessed 15 9 2022].

Annex 1: Dyed samples

(All images are Face (L) Back (R) of own images)

aNIR® Dianix® Rubine XF2 (Setup 1)



Figure 74: aNIR® Dianix® Rubine XF2 pale shade (setup 1)



Figure 75: aNIR® Dianix® Rubine XF2 pale shade (setup 1)



Figure 76: aNIR® Dianix® Rubine XF2 dark shade (setup 1)

aNIR® Dianix® Rubine CC (Setup 2)

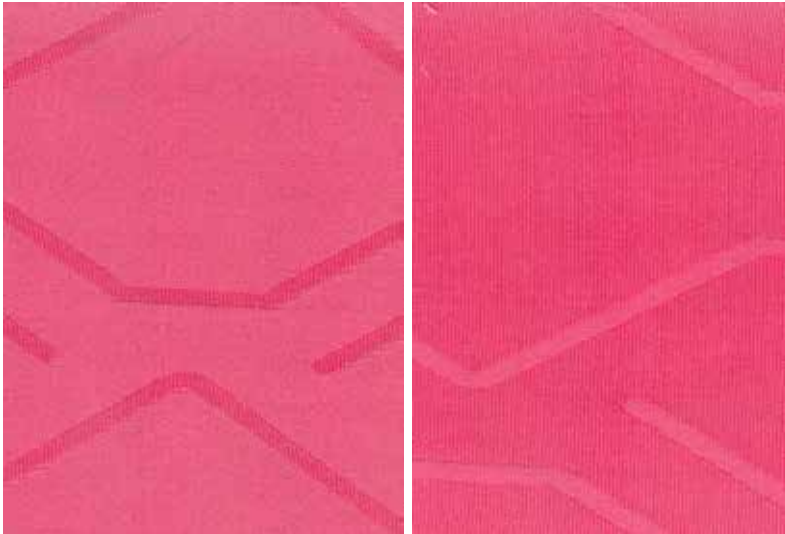


Figure 77: aNIR® Dianix® Rubine CC pale shade (setup 2)

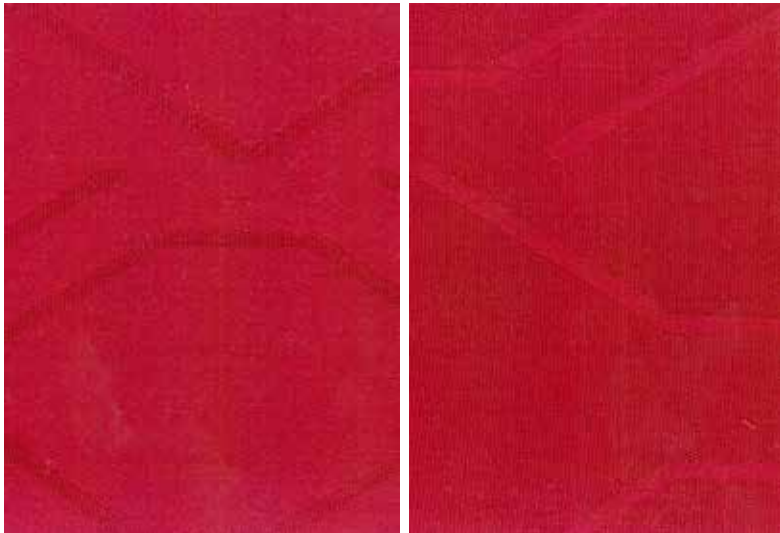


Figure 79: aNIR® Dianix® Rubine CC medium shade (setup 2)



Figure 78: aNIR® Dianix® Rubine CC dark shade (setup 2)

aNIR® Dianix® Yellow Brown XF2 (Setup 1)

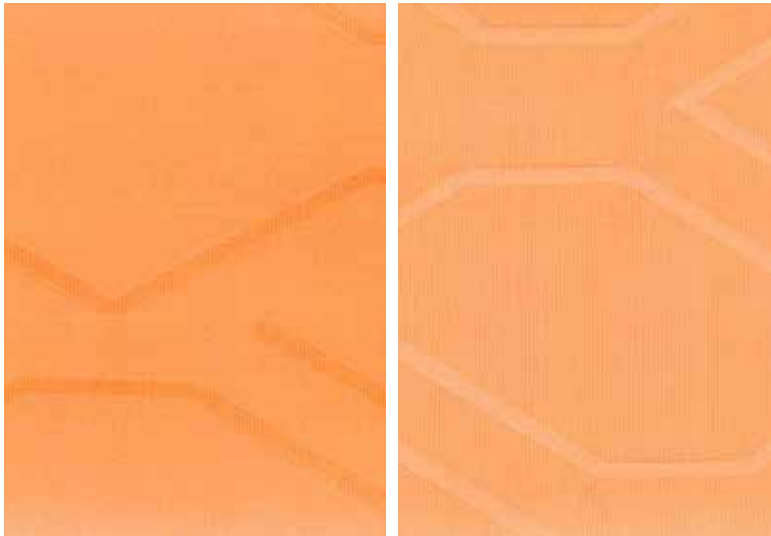


Figure 80: aNIR® Dianix® Yellow Brown XF2 pale shade (setup 1)

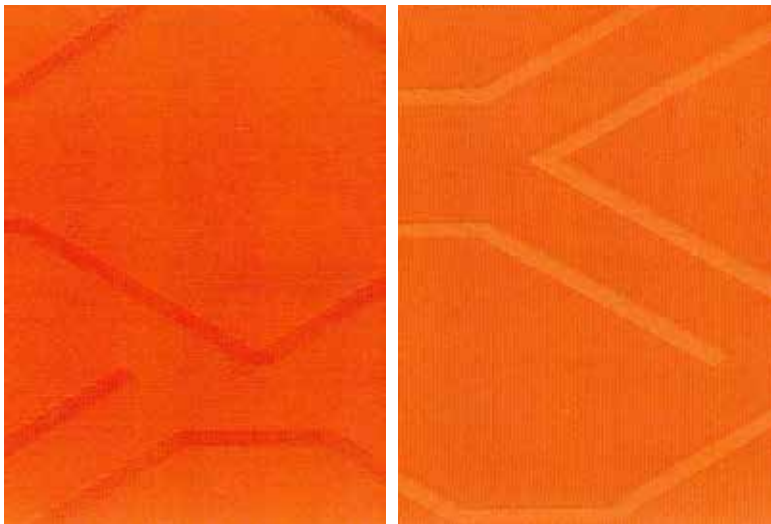


Figure 81: aNIR® Dianix® Yellow Brown XF2 medium shade (setup 1)

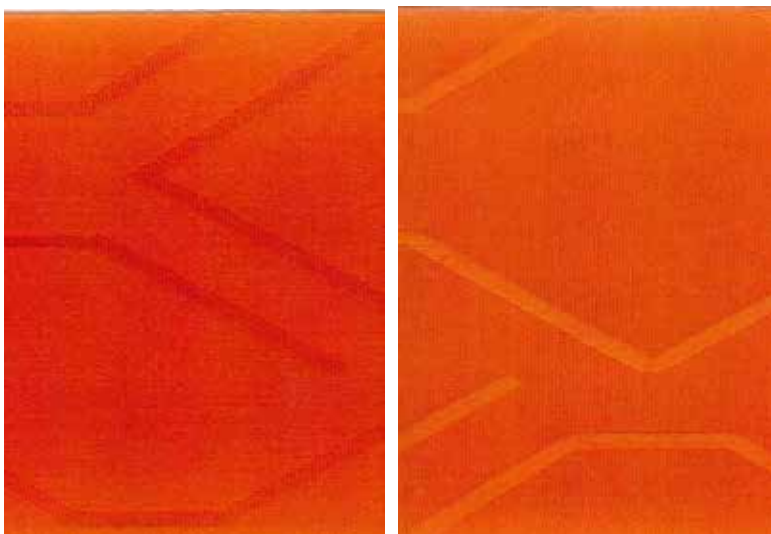


Figure 82: aNIR® Dianix® Yellow Brown XF2 dark shade (setup 1)

aNIR® Dianix® Yellow Brown XF2 (Setup 2)

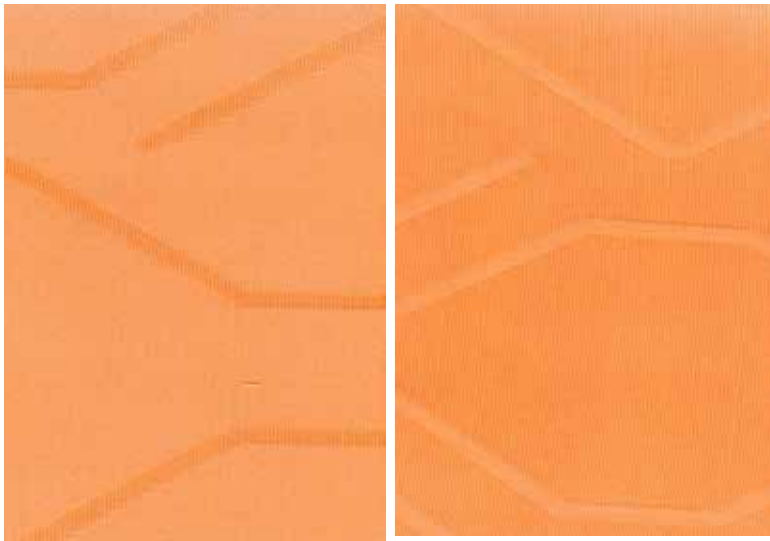


Figure 83: aNIR® Dianix® Yellow Brown XF2 pale shade (setup 2)

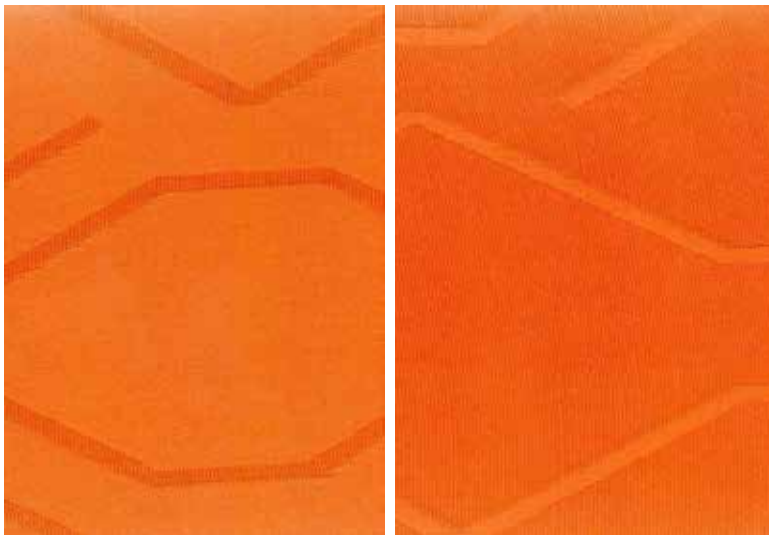


Figure 84: aNIR® Dianix® Yellow Brown XF2 medium shade (setup 2)

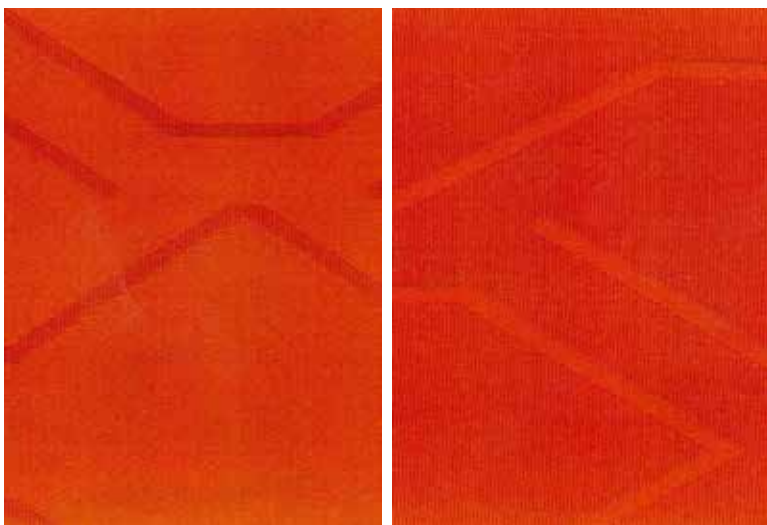


Figure 85: aNIR® Dianix® Yellow Brown XF2 dark shade (setup 2)

aNIR[®] Dianix[®] Yellow Brown CC (Setup 2)

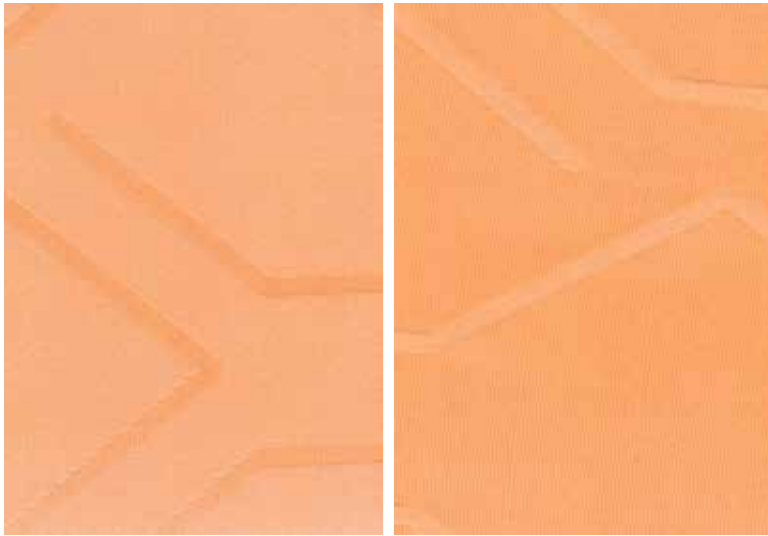


Figure 86: aNIR[®] Dianix[®] Yellow Brown CC pale shade (setup 2)

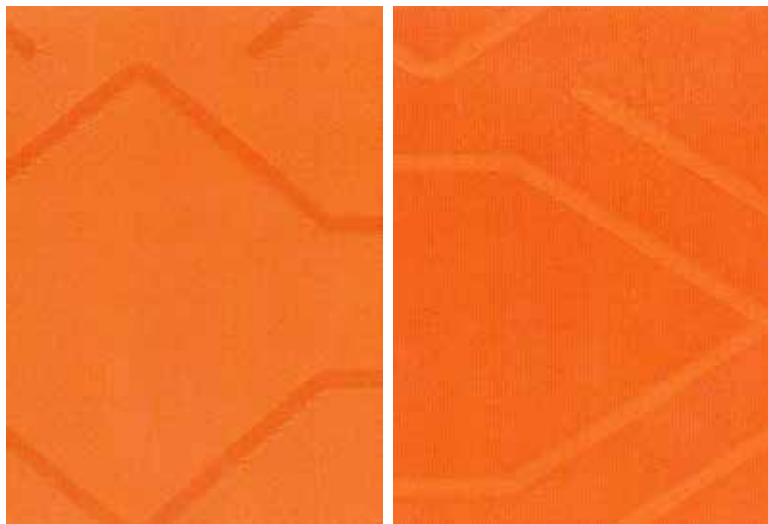


Figure 87: aNIR[®] Dianix[®] Yellow Brown CC medium shade (setup 2)

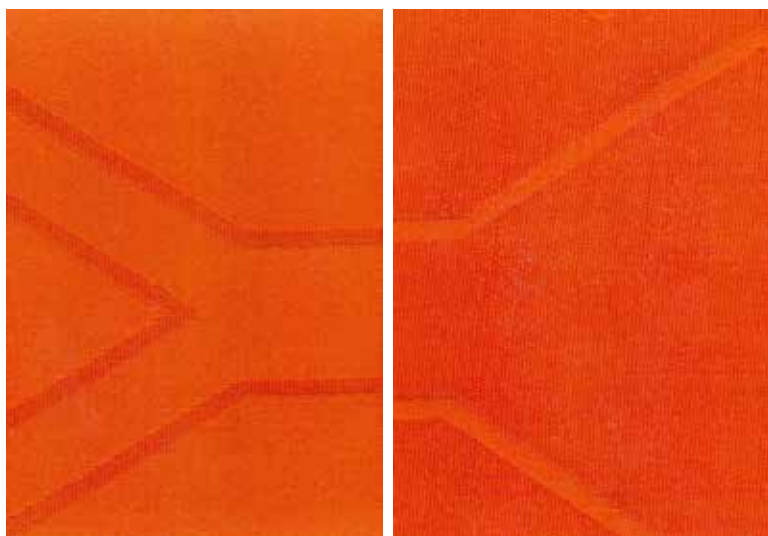


Figure 88: aNIR[®] Dianix[®] Yellow Brown CC dark shade (setup 2)

aNIR® Dianix® Navy XF2 (Setup 1)

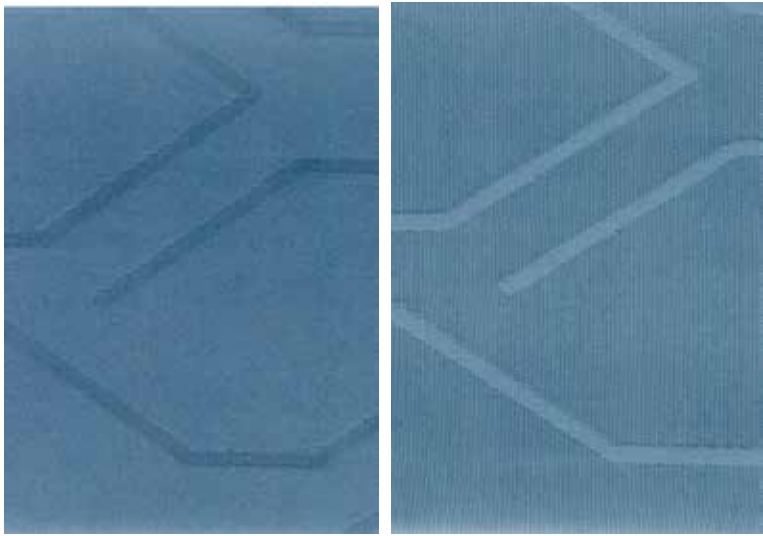


Figure 89: aNIR® Dianix® Navy XF2 pale shade (setup 1)

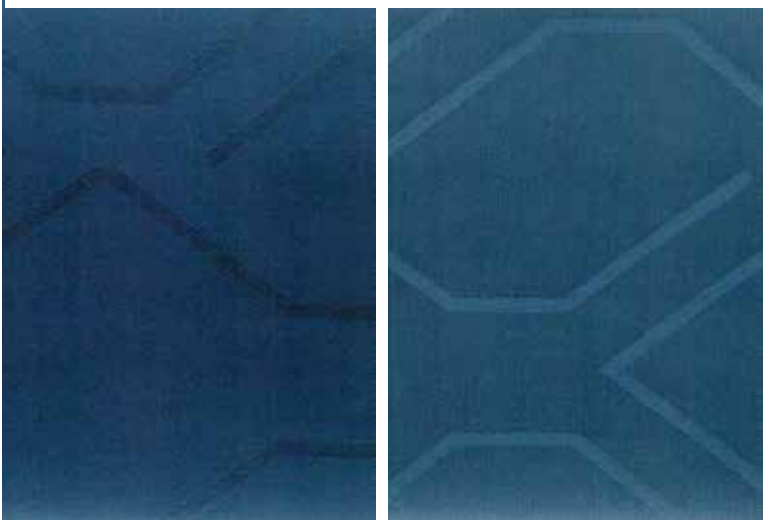


Figure 90: aNIR® Dianix® Navy XF2 medium shade (setup 1)

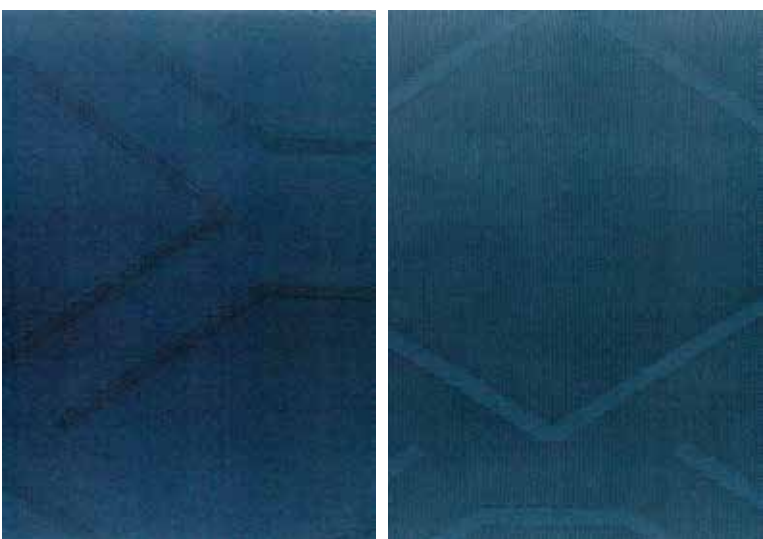


Figure 91: aNIR® Dianix® Navy XF2 dark shade (setup 1)

aNIR® Dianix® Navy XF2 (Setup 2)

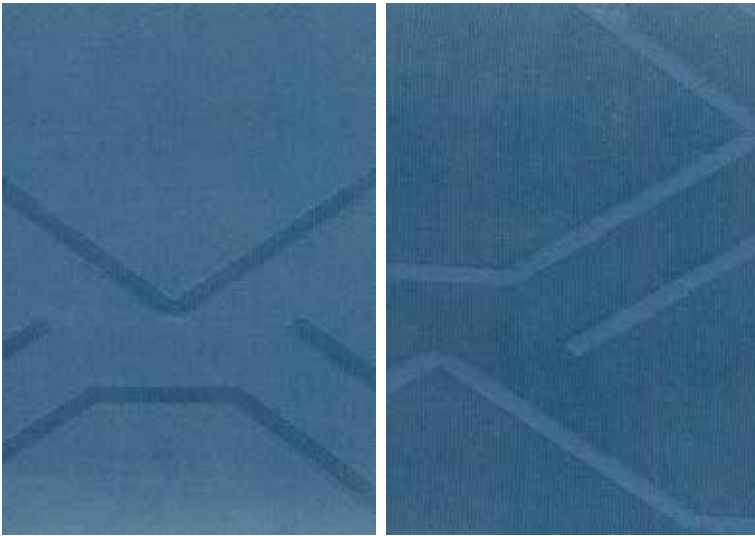


Figure 92: aNIR® Dianix® Navy XF2 pale shade (setup 2)



Figure 93: aNIR® Dianix® Navy XF2 medium shade (setup 2)



Figure 94: aNIR® Dianix® Navy XF2 dark shade (setup 2)

aNIR® Dianix® Navy CC (Setup 2)

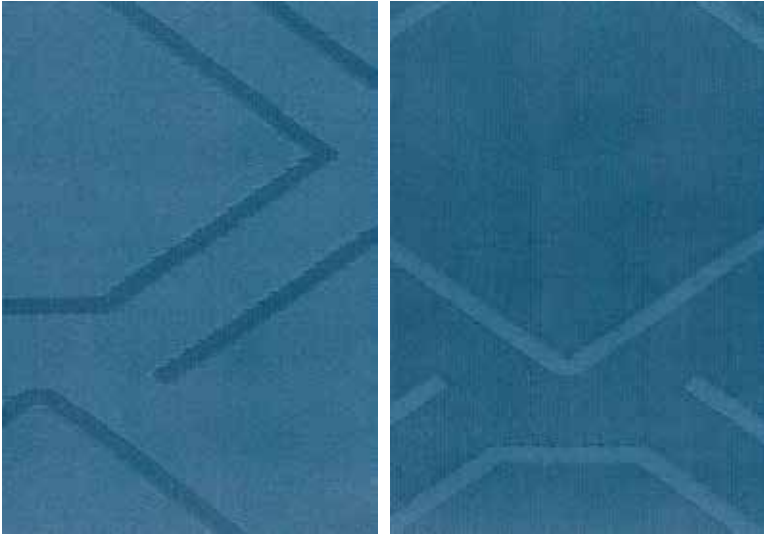


Figure 95: aNIR® Dianix® Navy CC pale shade (setup 2)

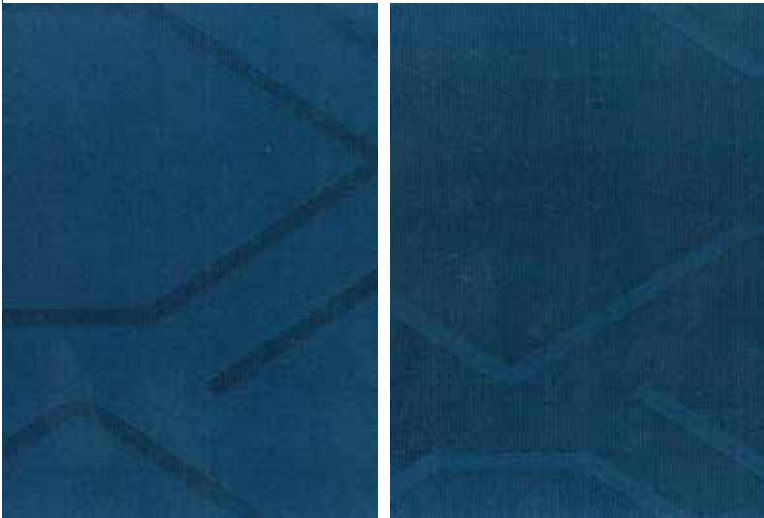


Figure 96: aNIR® Dianix® Navy CC medium shade (setup 2)



Figure 97: aNIR® Dianix® Navy CC dark shade (setup 2)

aNIR® Dianix® Black 300% XF2 (Setup 1)

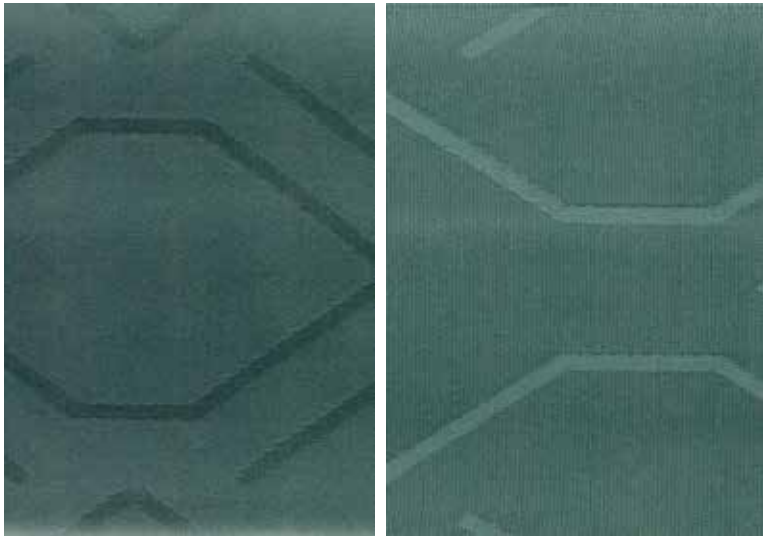


Figure 98: aNIR® Dianix® Black 300% XF2 pale shade (setup 1)

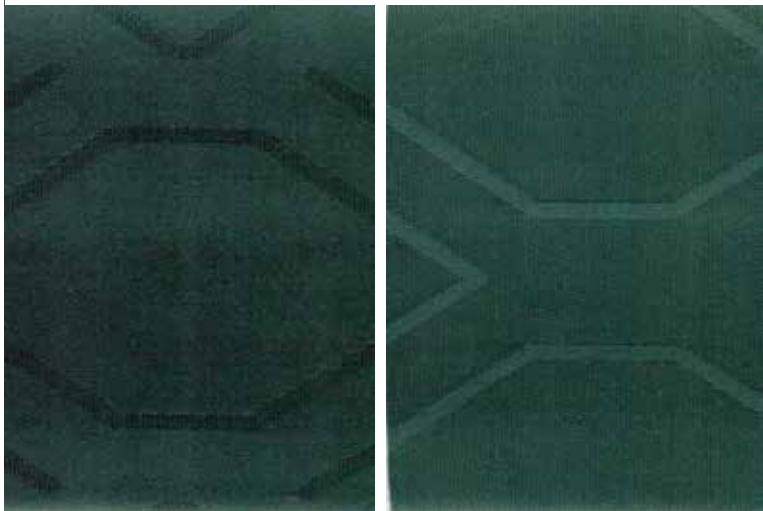


Figure 99: aNIR® Dianix® Black 300% XF2 medium shade (setup 1)

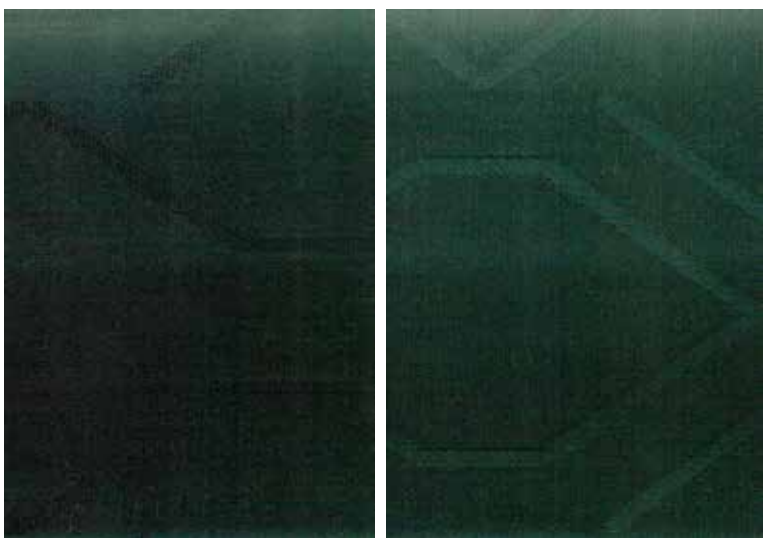


Figure 100: aNIR® Dianix® Black 300% XF2 dark shade (setup 1)

aNIR[®] Dianix[®] Black 300% XF2 (Setup 2)



Figure 101: aNIR[®] Dianix[®] Black 300% XF2 pale shade (setup 2)



Figure 102: aNIR[®] Dianix[®] Black 300% XF2 medium shade (setup 2)



Figure 103: aNIR[®] Dianix[®] Black 300% XF2 dark shade (setup 2)

aNIR® Dianix® Black CC-R (Setup 2)

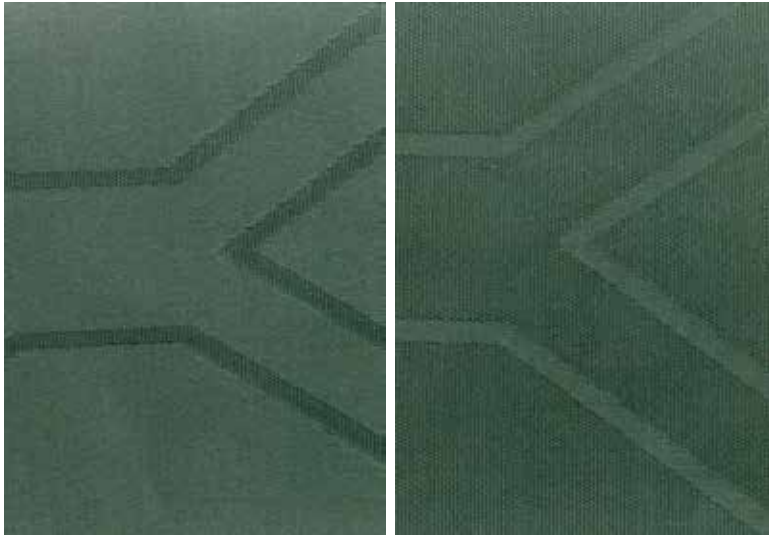


Figure 104: aNIR® Dianix® Black CC-R pale shade (setup 2)



Figure 105: aNIR® Dianix® Black CC-R medium shade (setup 2)

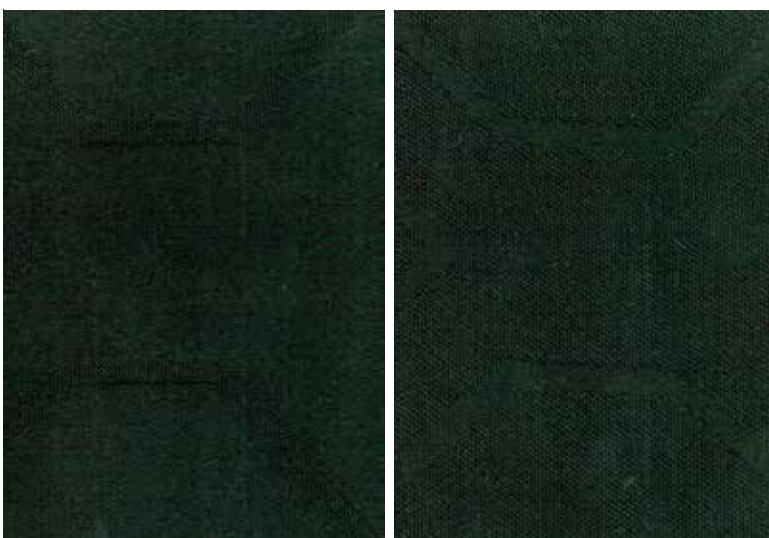


Figure 106: aNIR® Dianix® Black CC-R dark shade (setup 2)

Annex 2: Color fastness to wash (ISO 105 F10)
(All images are Face(L) of own pictures)



Figure 108: Convection process Dianix® Rubine XF2 pale shade



Figure 109: Convection process Dianix® Rubine XF2 medium shade



Figure 107: Convection process Dianix® Rubine XF2 dark shade

sample after washing

original multi fiber strip



Figure 110: aNIR[®] Dianix[®] Rubine XF2 pale shade (Setup 1)



Figure 111: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 1)



Figure 112: aNIR[®] Dianix[®] Rubine XF2 dark shade (Setup 1)



Figure 115: aNIR[®] Dianix[®] Rubine XF2 pale shade (Setup 2)



Figure 113: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 2)



Figure 114: aNIR[®] Dianix[®] Rubine XF2 dark shade (Setup 2)



Figure 116: aNIR[®] Dianix[®] Rubine CC pale shade (Setup 2)



Figure 117: aNIR[®] Dianix[®] Rubine CC medium shade (Setup 2)



Figure 118: aNIR[®] Dianix[®] Rubine CC dark shade (Setup 2)



Figure 120: convection process Dianix® Yellow Brown XF2 pale shade



Figure 121: convection process Dianix® Yellow Brown XF2 medium shade



Figure 119: convection process Dianix® Yellow Brown XF2 dark shade



Figure 123: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 1)



Figure 122: aNIR[®] Dianix[®] Yellow Brown XF2 medium shade (Setup 1)



Figure 124: aNIR[®] Dianix[®] Yellow Brown XF2 dark shade (Setup 1)



Figure 125: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 2)



Figure 126: aNIR[®] Dianix[®] Yellow Brown XF2 medium shade (Setup 2)



Figure 127: aNIR[®] Dianix[®] Yellow Brown XF2 dark shade (Setup 2)



Figure 128: aNIR[®] Dianix[®] Yellow Brown CC dark shade (Setup 2)



Figure 129: aNIR[®] Dianix[®] Yellow Brown CC dark shade (Setup 2)



Figure 130: aNIR[®] Dianix[®] Yellow Brown CC dark shade (Setup 2)



Figure 131: convection process Dianix® Navy XF2 pale shade



Figure 132: convection process Dianix® Navy XF2 medium shade



Figure 133: convection process Dianix® Navy XF2 dark shade

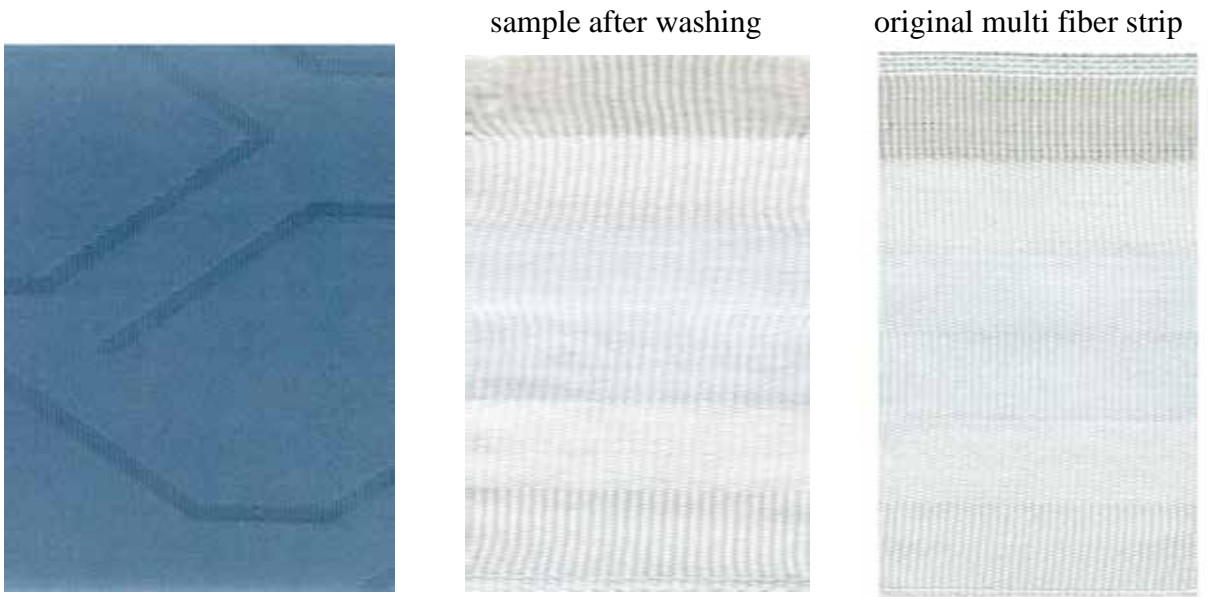


Figure 134: aNIR[®] Dianix[®] Navy XF2 pale shade (Setup 1)



Figure 135: aNIR[®] Dianix[®] Navy XF2 medium shade (Setup 1)



Figure 136: aNIR[®] Dianix[®] Navy XF2 dark shade (Setup 1)

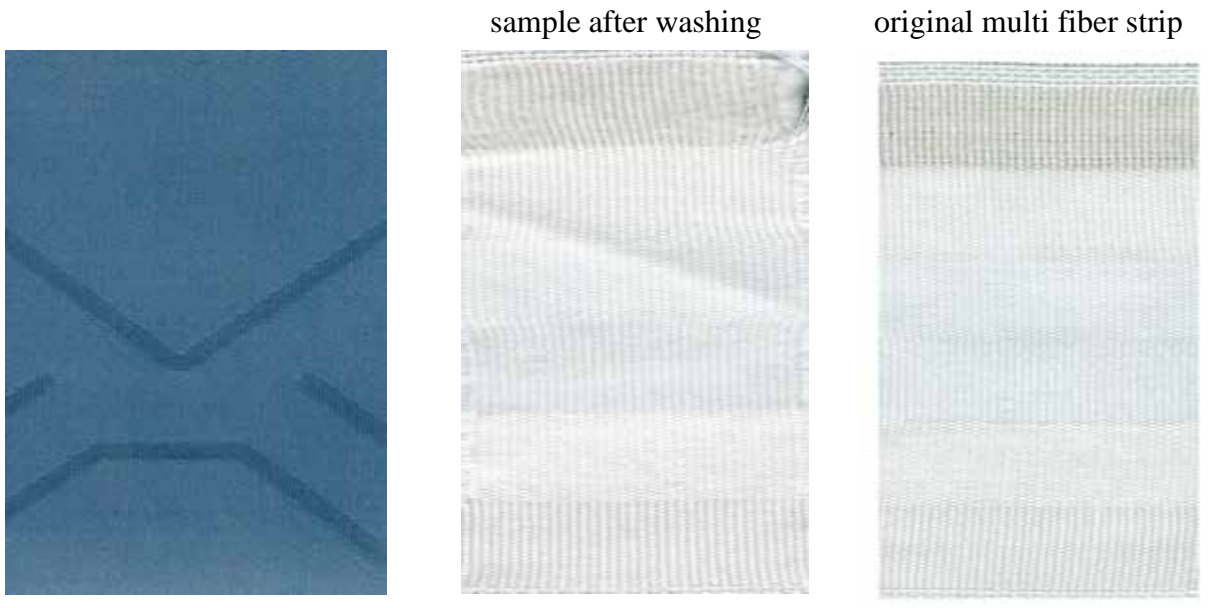


Figure 137: aNIR® Dianix® Navy XF2 pale shade (Setup 2)



Figure 138: aNIR® Dianix® Navy XF2 medium shade (Setup 2)



Figure 139: aNIR® Dianix® Navy XF2 dark shade (Setup 2)

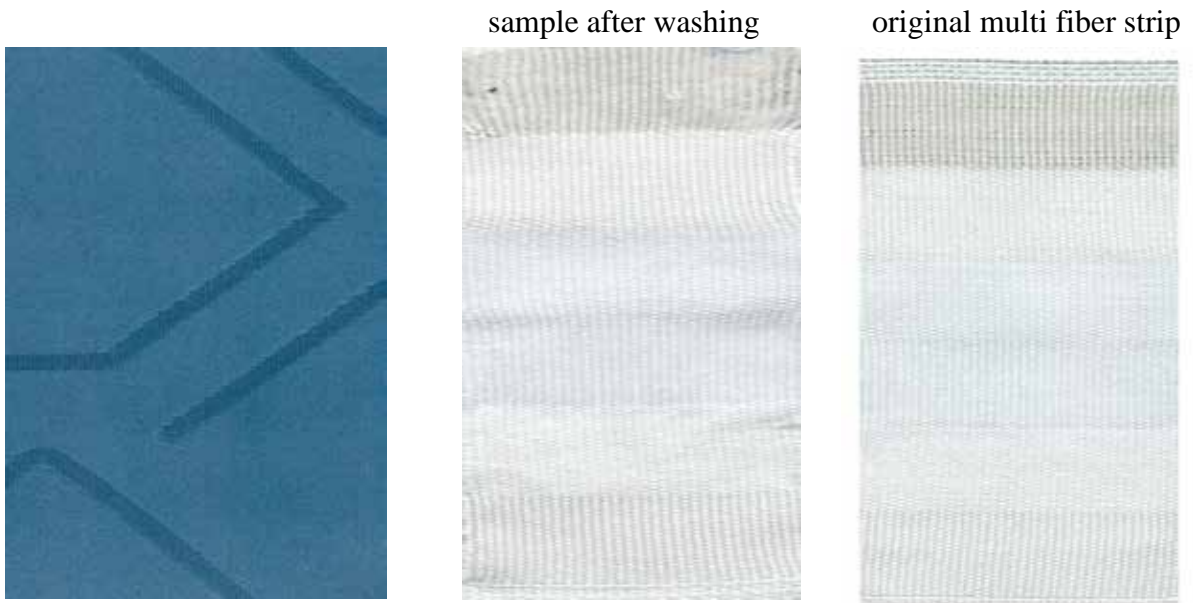


Figure 140: aNIR[®] Dianix[®] Navy CC pale shade (Setup 2)



Figure 141: aNIR[®] Dianix[®] Navy CC medium shade (Setup 2)



Figure 142: aNIR[®] Dianix[®] Navy CC dark shade (Setup 2)



Figure 143: convection process Dianix® Black XF2 300 % pale shade



Figure 144: convection process Dianix® Black XF2 300 % medium shade



Figure 145: convection process Dianix® Black XF2 300 % dark shade

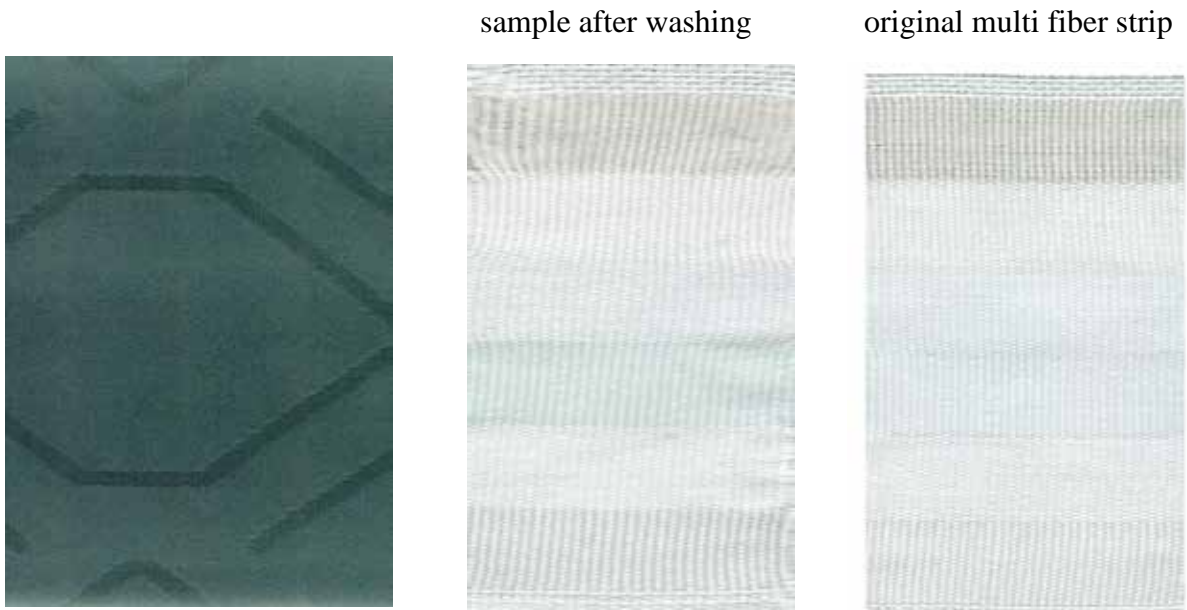


Figure 146: aNIR[®] Dianix[®] Black XF2 300 % pale shade (Setup 1)



Figure 147: aNIR[®] Dianix[®] Black XF2 300 % medium shade (Setup 1)



Figure 148: aNIR[®] Dianix[®] Black XF2 300 % dark shade (Setup 1)



Figure 149: aNIR[®] Dianix[®] Black XF2 300 % pale shade (Setup 2)



Figure 150: aNIR[®] Dianix[®] Black XF2 300 % medium shade (Setup 2)



Figure 151: aNIR[®] Dianix[®] Black XF2 300 % dark shade (Setup 2)



Figure 152: aNIR[®] Dianix[®] Black CC-R pale shade (Setup 2)



Figure 153: aNIR[®] Dianix[®] Black CC-R medium shade (Setup 2)



Figure 154: aNIR[®] Dianix[®] Black CC-R dark shade (Setup 2)

**Annex 3: Color fastness to dry heat (ISO 105-
P01:1993(E))**

(All images are Face(L) of own pictures)

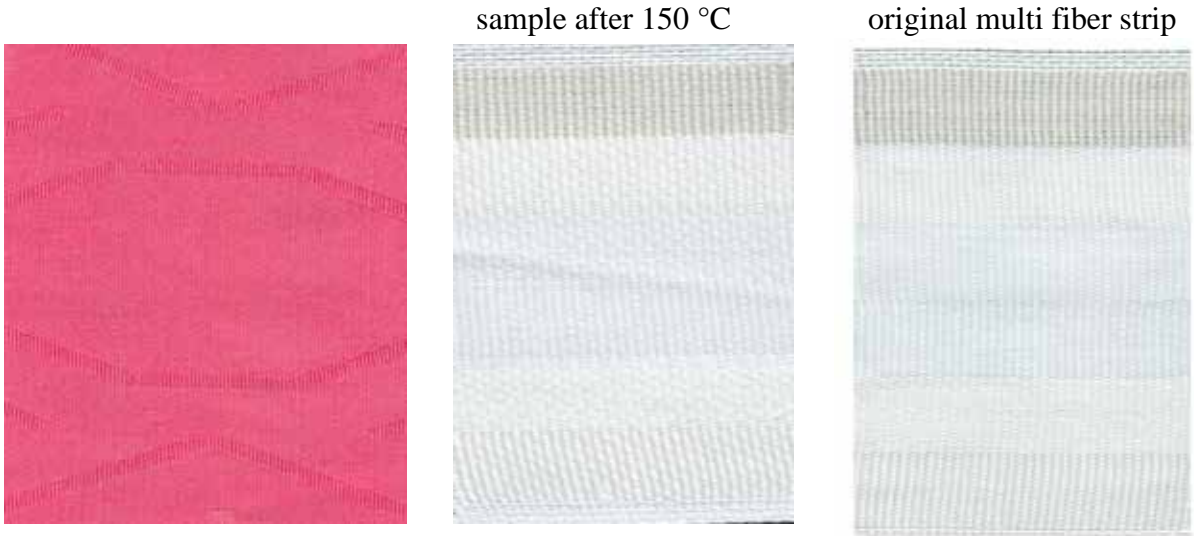


Figure 155: convection process Dianix® Rubine XF2 pale shade sample after 150 °C



Figure 156: convection process Dianix® Rubine XF2 pale shade sample after 180 °C



Figure 157: convection process Dianix® Rubine XF2 pale shade sample after 210 °C



Figure 158: convection process Dianix® Rubine medium shade sample after 150 °C



Figure 159: convection process Dianix® Rubine medium shade sample after 180 °C



Figure 160: convection process Dianix® Rubine medium shade sample after 210 °C



Figure 161: convection process Dianix® Rubine XF2 dark shade sample after 150 °C



Figure 162: convection process Dianix® Rubine XF2 dark shade sample after 180 °C



Figure 163: convection process Dianix® Rubine XF2 dark shade sample after 210 °C



Figure 164: aNIR[®] Dianix[®] Rubine XF2 pale shade (Setup 1) sample after 150 °C

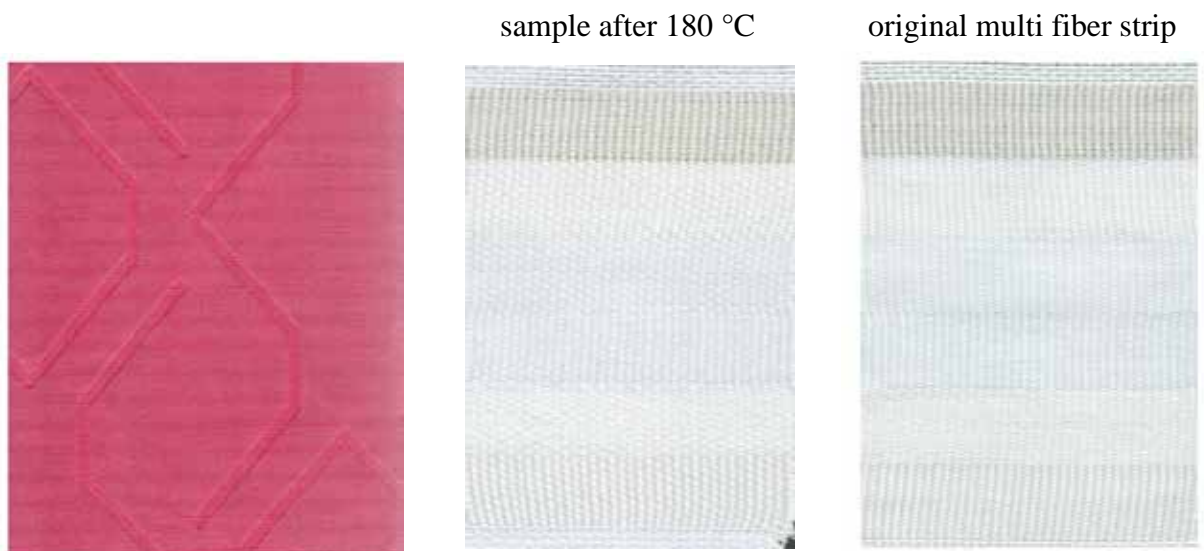


Figure 165: aNIR[®] Dianix[®] Rubine XF2 pale shade (Setup 1) sample after 180 °C



Figure 166: aNIR[™] Dianix[™] Rubine XF2 pale shade (Setup 1) sample after 210 °C

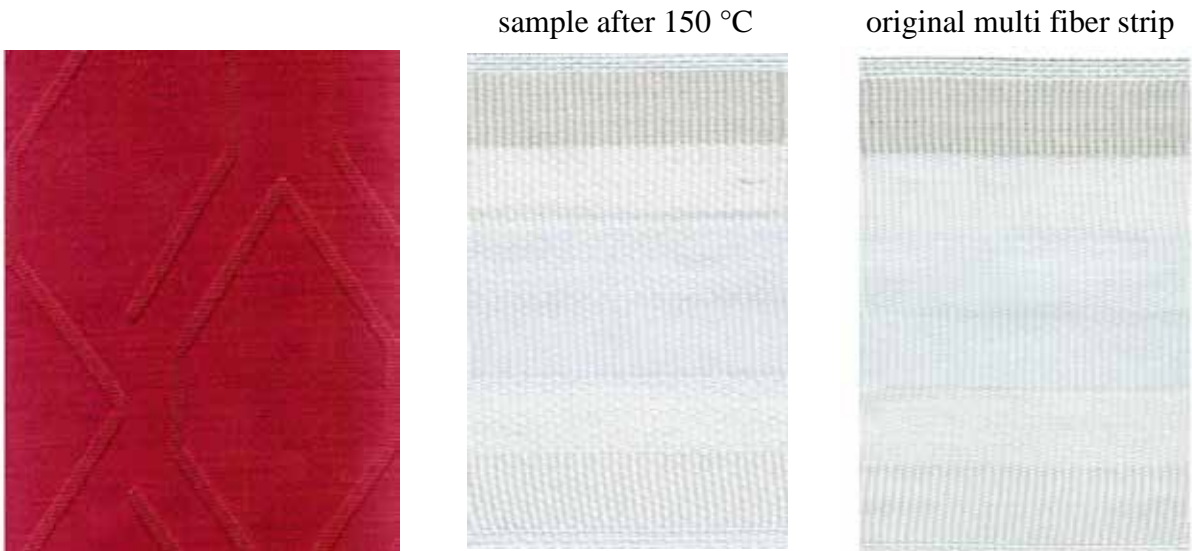


Figure 167: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 1) sample after 150 °C



Figure 168: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 1) sample after 180 °C



Figure 169: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 1) sample after 210 °C

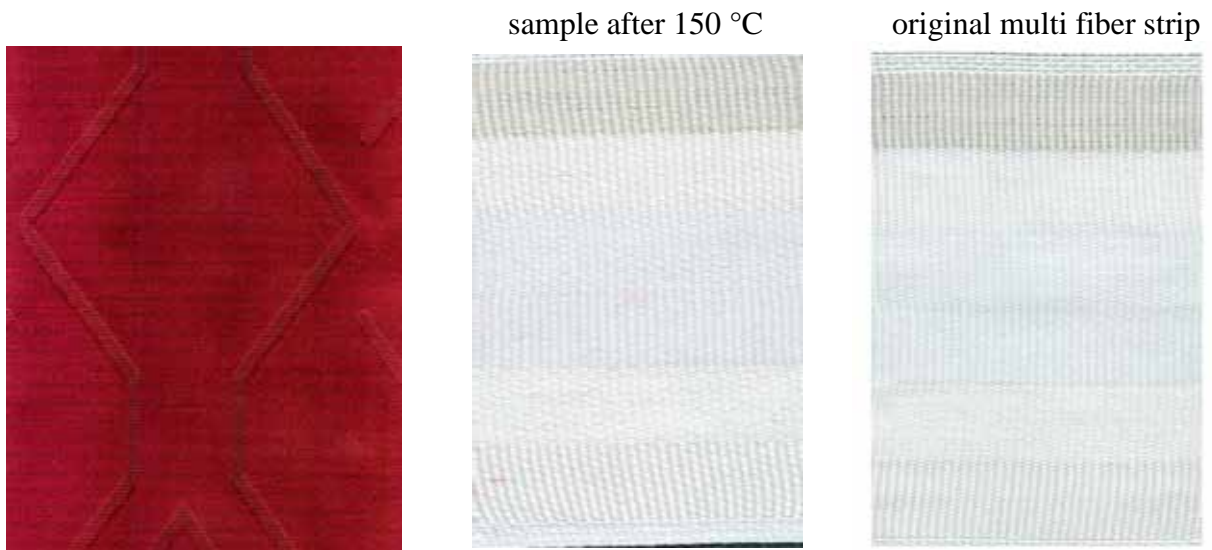


Figure 170: aNIR[®] Dianix[®] Rubine XF2 dark shade (Setup 1) sample after 150 °C



Figure 171: aNIR[®] Dianix[®] Rubine XF2 dark shade (Setup 1) sample after 180 °C



Figure 172: aNIR[®] Dianix[®] Rubine XF2 dark shade (Setup 1) sample after 210 °C



Figure 173: aNIR® Dianix® Rubine XF2 pale shade (Setup 2) sample after 150 °C



Figure 174: aNIR® Dianix® Rubine XF2 pale shade (Setup 2) sample after 180 °C

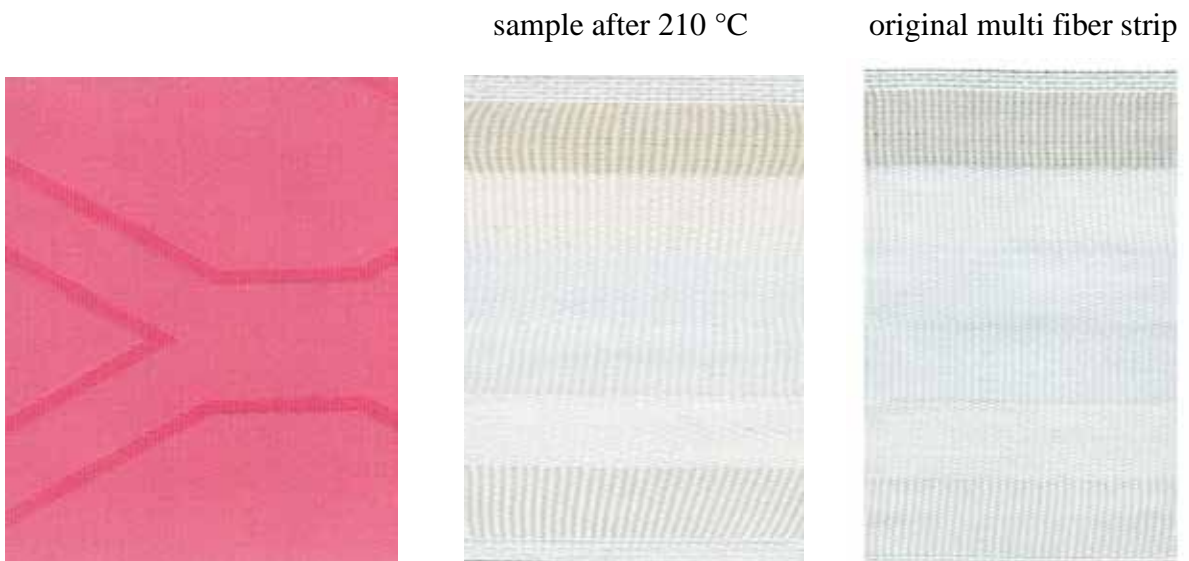


Figure 175: aNIR® Dianix® Rubine XF2 pale shade (Setup 2) sample after 210 °C



Figure 176: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 2) sample after 150 °C



Figure 177: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 2) sample after 180 °C



Figure 178: aNIR[®] Dianix[®] Rubine XF2 medium shade (Setup 2) sample after 210 °C



Figure 179: aNIR® Dianix® Rubine XF2 dark shade (Setup 2) sample after 150 °C



Figure 180: aNIR® Dianix® Rubine XF2 dark shade (Setup 2) sample after 180 °C

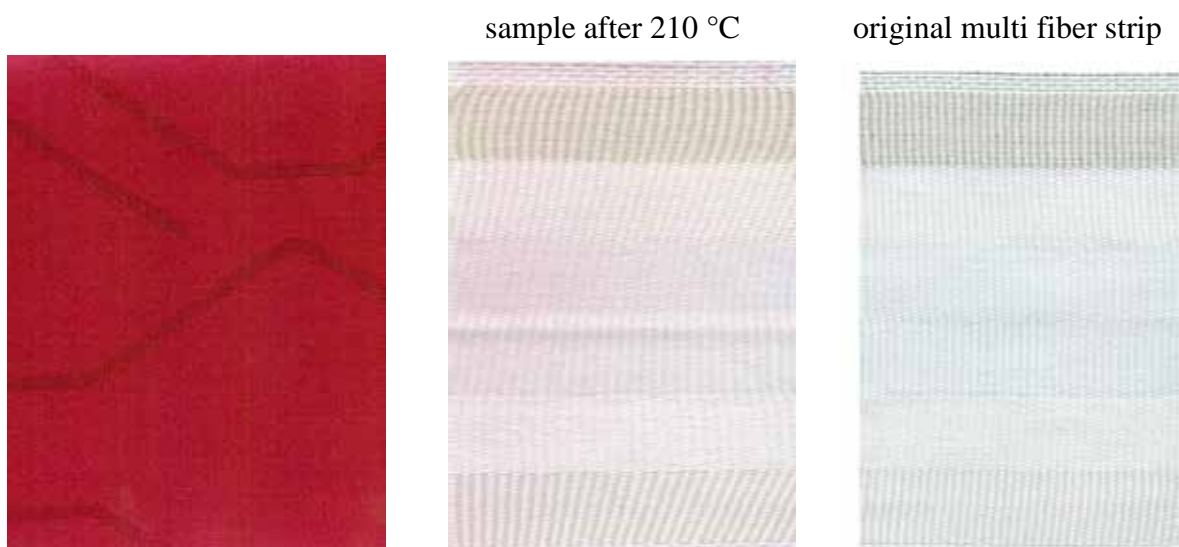


Figure 181: aNIR® Dianix® Rubine XF2 dark shade (Setup 2) sample after 210 °C

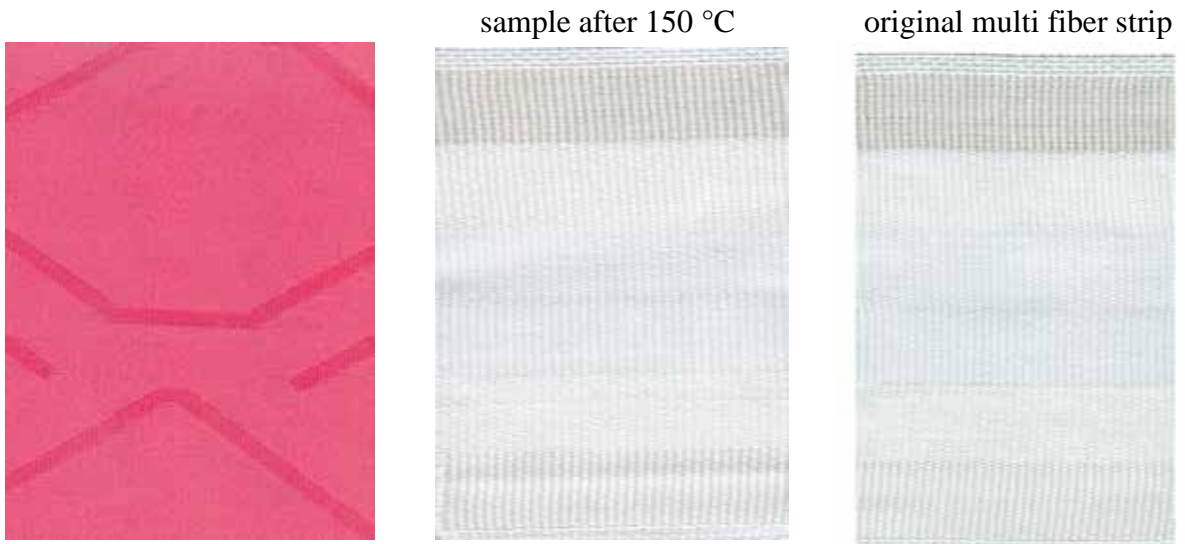


Figure 182: aNIR[®] Dianix[®] Rubine CC pale shade (Setup 2) sample after 150 °C



Figure 183: aNIR[®] Dianix[®] Rubine CC pale shade (Setup 2) sample after 180 °C



Figure 184: aNIR[®] Dianix[®] Rubine CC pale shade (Setup 2) sample after 210 °C



Figure 185: aNIR® Dianix® Rubine CC medium shade (Setup 2) sample after 150 °C



Figure 186: aNIR® Dianix® Rubine CC medium shade (Setup 2) sample after 180 °C



Figure 187: aNIR® Dianix® Rubine CC medium shade (Setup 2) sample after 210 °C



Figure 188: aNIR[®] Dianix[®] Rubine CC dark shade (Setup 2) sample after 150 °C



Figure 189: aNIR[®] Dianix[®] Rubine CC dark shade (Setup 2) sample after 180 °C

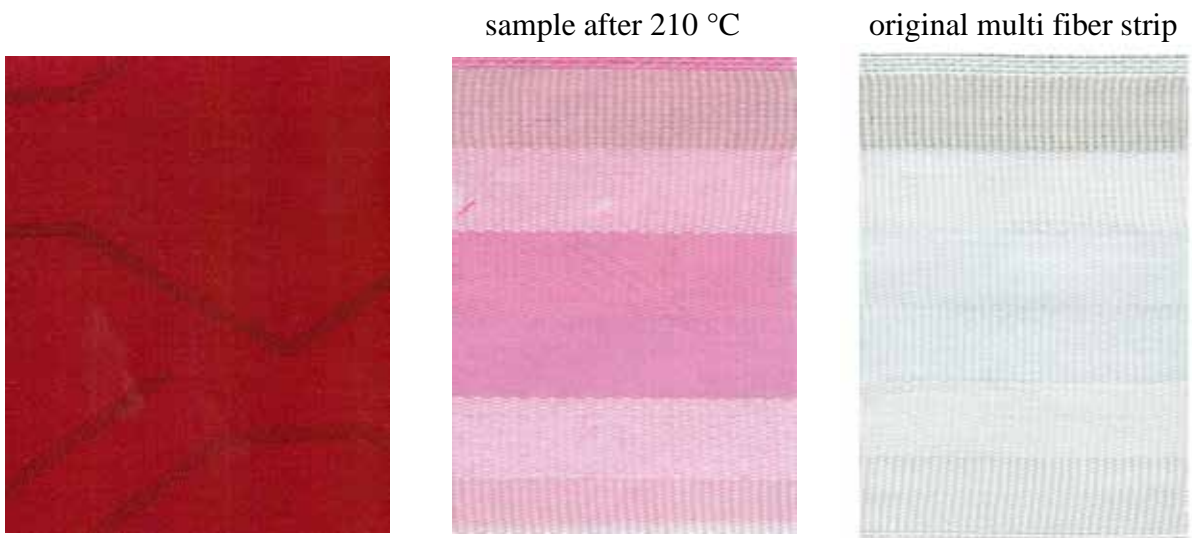


Figure 190: aNIR[®] Dianix[®] Rubine CC dark shade (Setup 2) sample after 210 °C



Figure 191: convection process Dianix® Yellow Brown pale shade sample after 150 °C



Figure 192: convection process Dianix® Yellow Brown pale shade sample after 180 °C



Figure 193: convection process Dianix® Yellow Brown pale shade sample after 210 °C



Figure 194: convection process Dianix® Yellow Brown medium shade sample after 150 °C

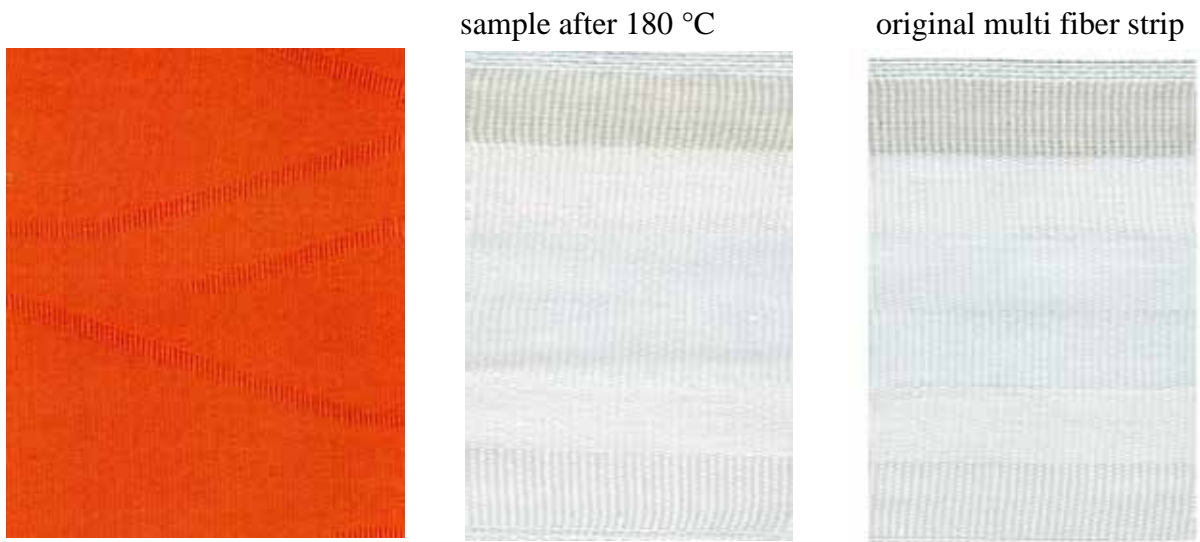


Figure 195: convection process Dianix® Yellow Brown medium shade sample after 180 °C

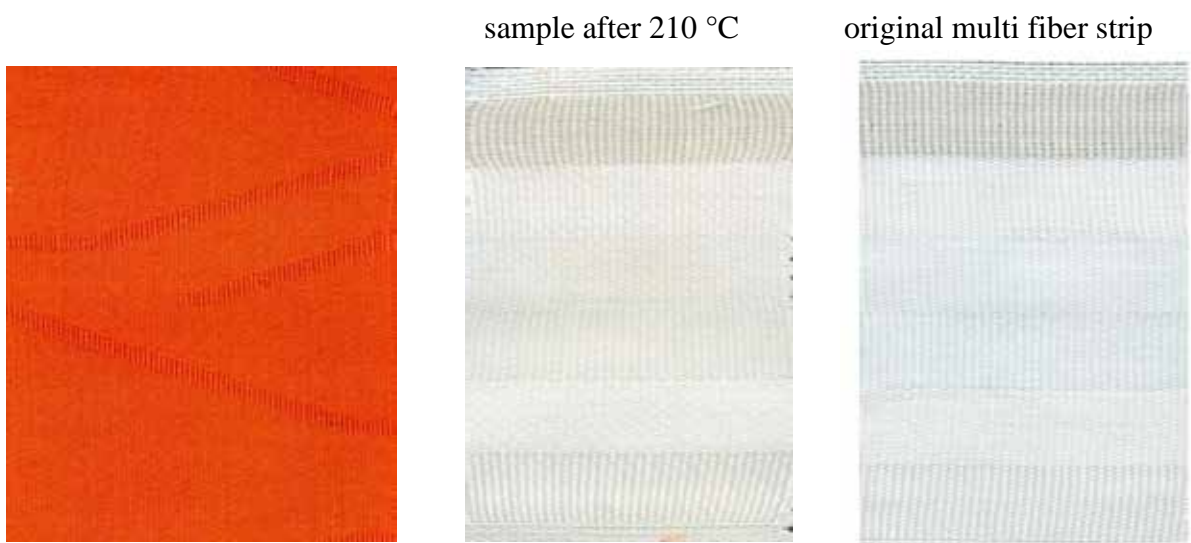


Figure 196: convection process Dianix® Yellow Brown medium shade sample after 210 °C

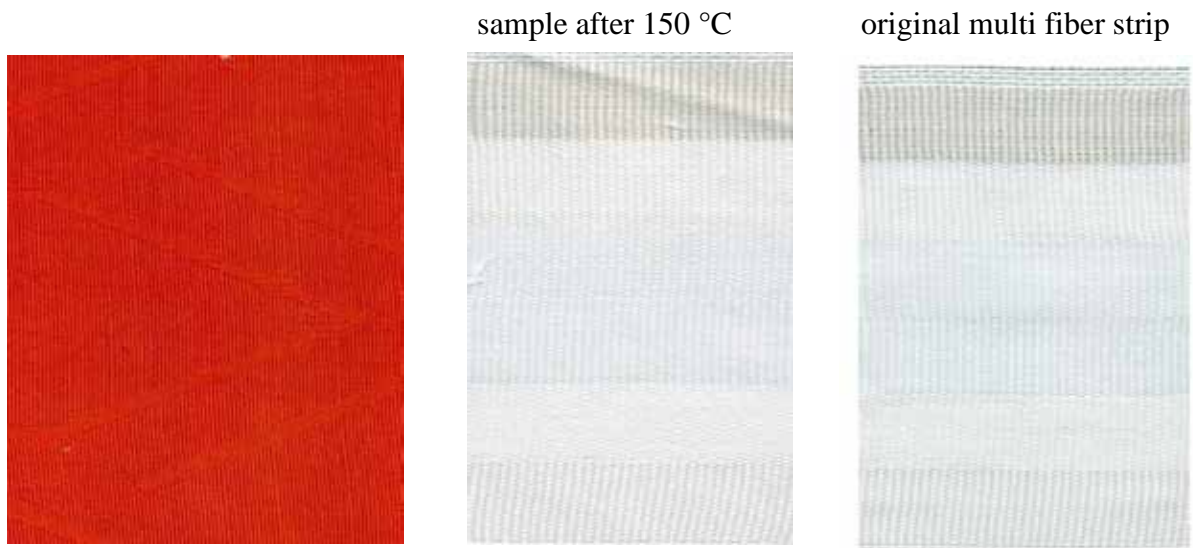


Figure 197: convection process Dianix® Yellow Brown dark shade sample after 150 °C



Figure 198: convection process Dianix® Yellow Brown dark shade sample after 180 °C



Figure 199: convection process Dianix® Yellow Brown dark shade sample after 210 °C



Figure 200: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 1) sample after 150 °C

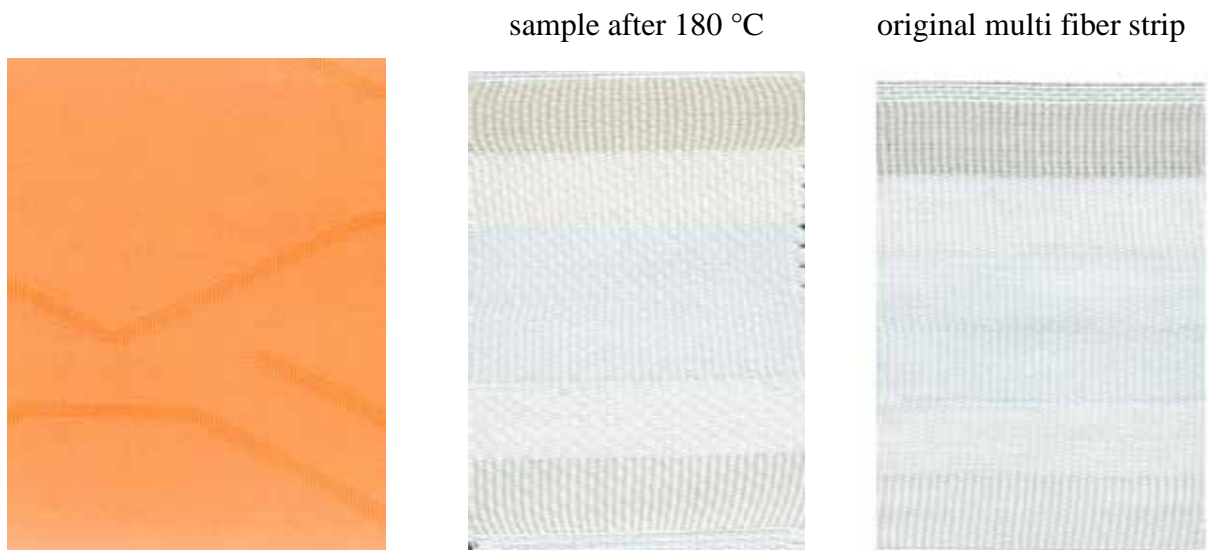


Figure 201: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 1) sample after 180 °C



Figure 202: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 1) sample after 210 °C

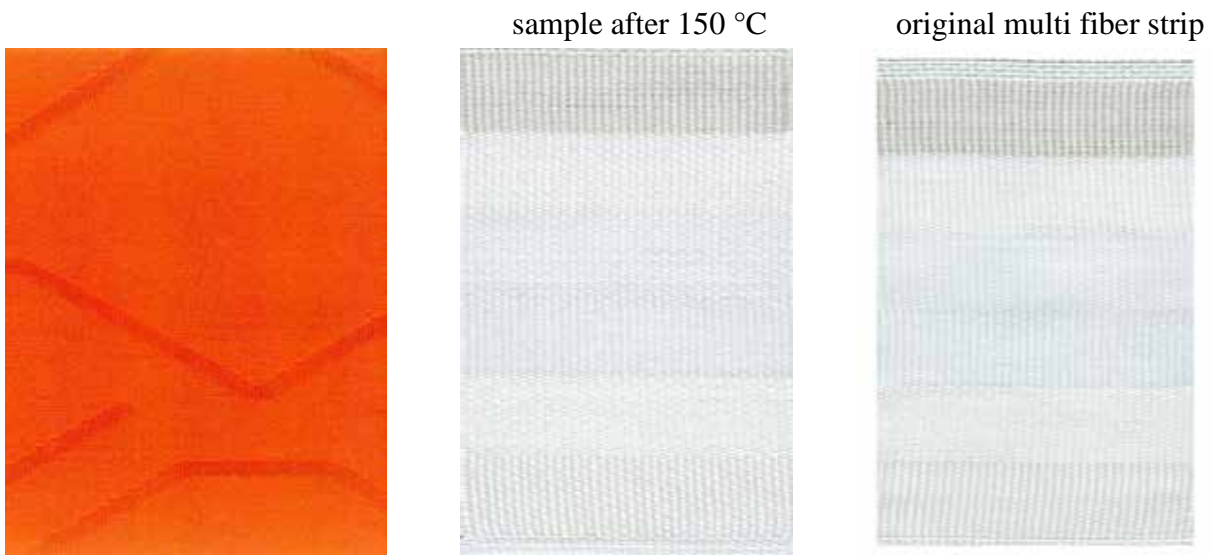


Figure 203: aNIR® Dianix® Yellow Brown XF2 medium shade (Setup 1) sample after 150 °C

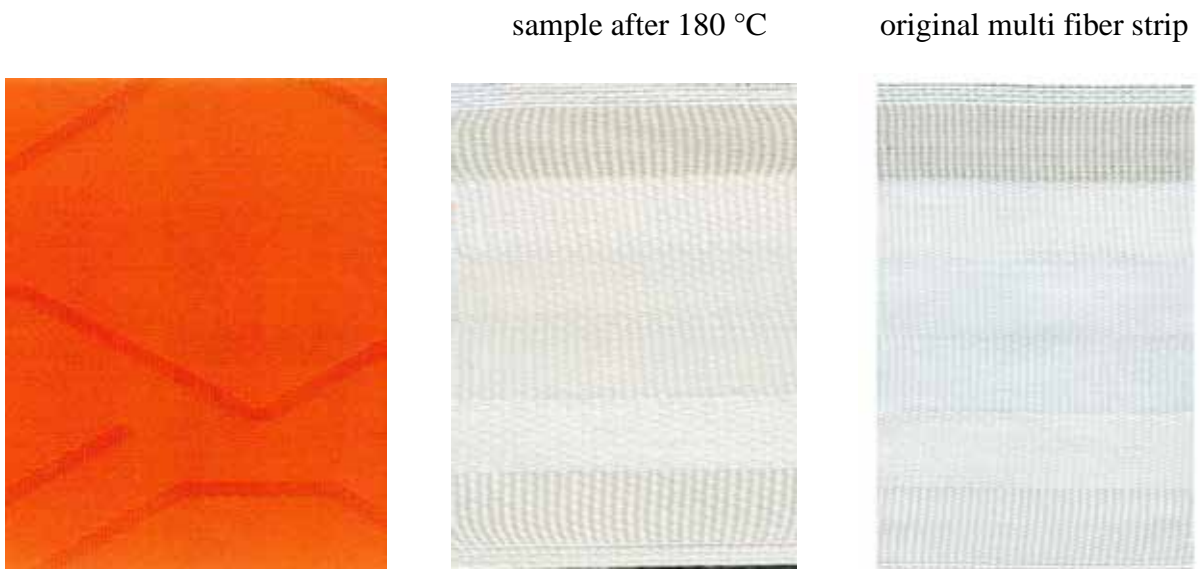


Figure 204: aNIR® Dianix® Yellow Brown XF2 medium shade (Setup 1) sample after 180 °C

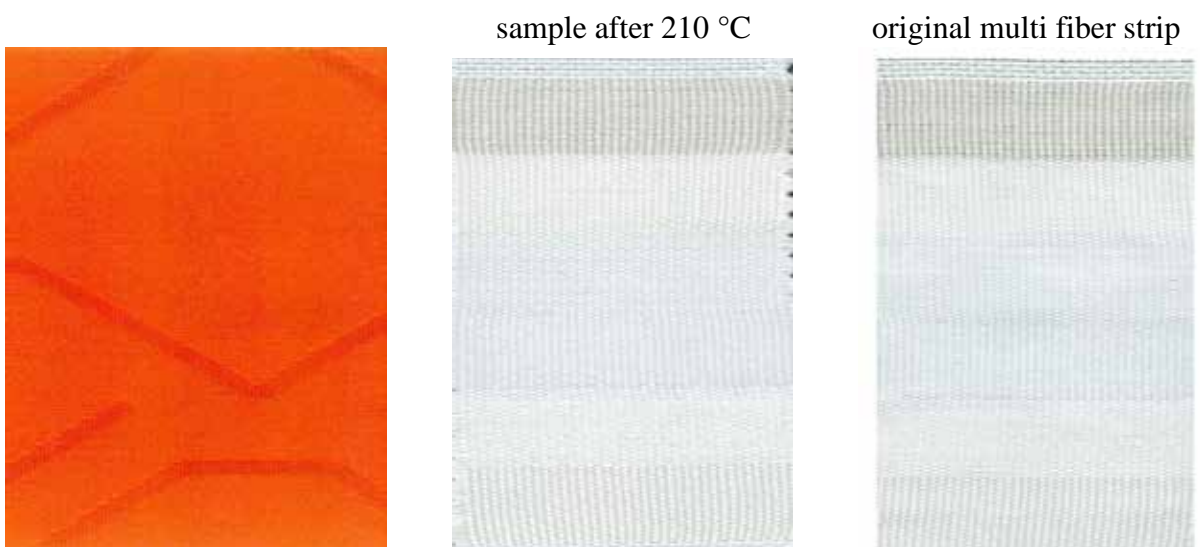


Figure 205: aNIR® Dianix® Yellow Brown XF2 medium shade (Setup 1) sample after 210 °C

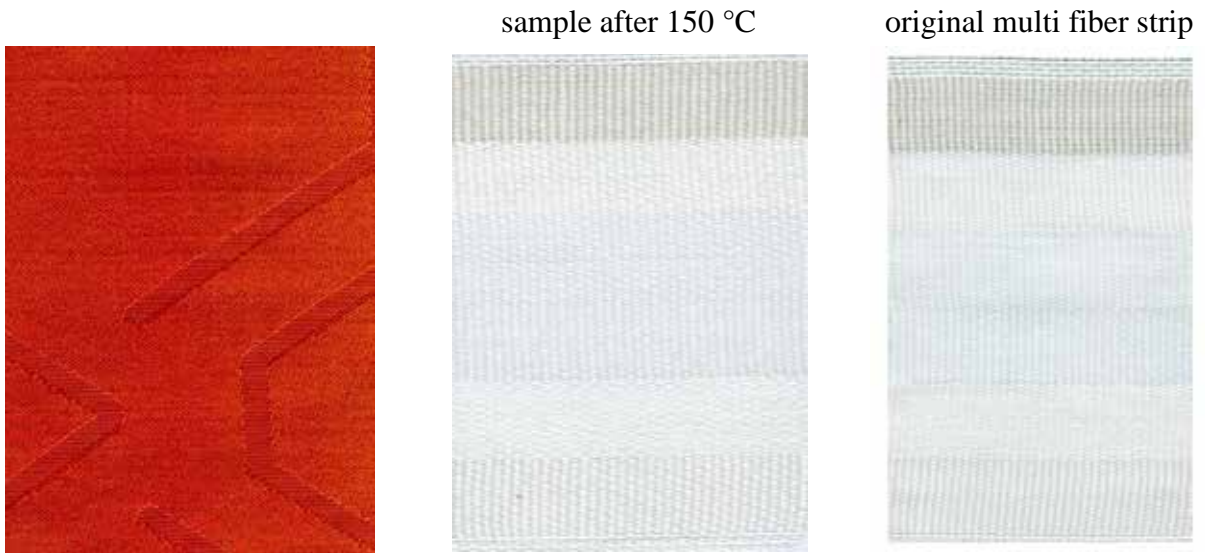


Figure 206: aNIR® Dianix® Yellow Brown XF2 dark shade (Setup 1) sample after 150 °C



Figure 207: aNIR® Dianix® Yellow Brown XF2 dark shade (Setup 1) sample after 180 °C



Figure 208: aNIR® Dianix® Yellow Brown XF2 dark shade (Setup 1) sample after 210 °C



Figure 209: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 2) sample after 150 °C



Figure 210: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 2) sample after 180 °C



Figure 211: aNIR[®] Dianix[®] Yellow Brown XF2 pale shade (Setup 2) sample after 210 °C

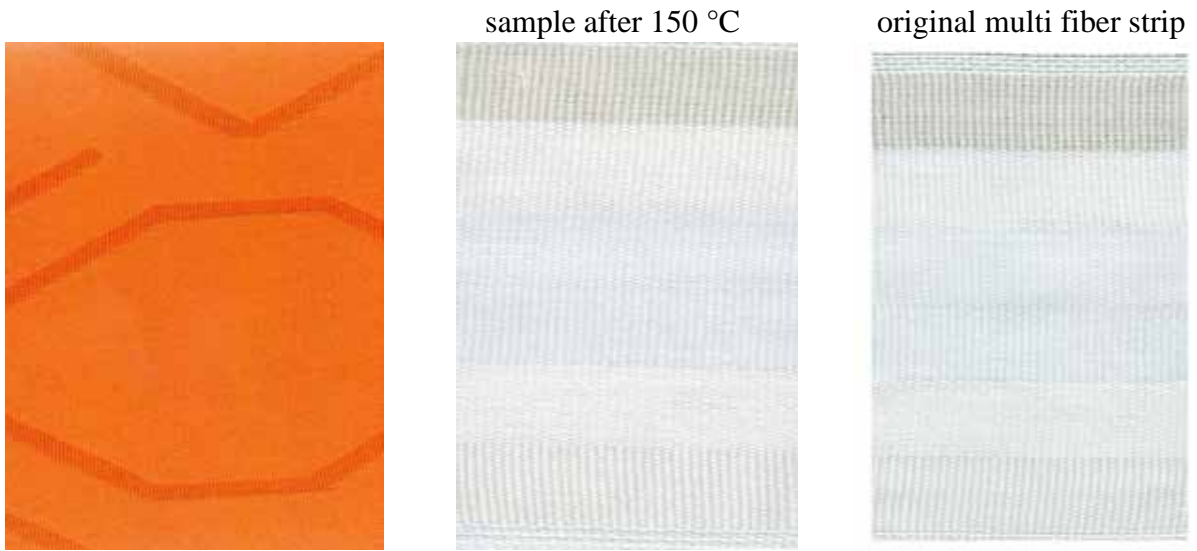


Figure 212: aNIR[®] Dianix[®] Yellow Brown XF2 medium shade (Setup 2) sample after 150 °C



Figure 213: aNIR[®] Dianix[®] Yellow Brown XF2 medium shade (Setup 2) sample after 180 °C

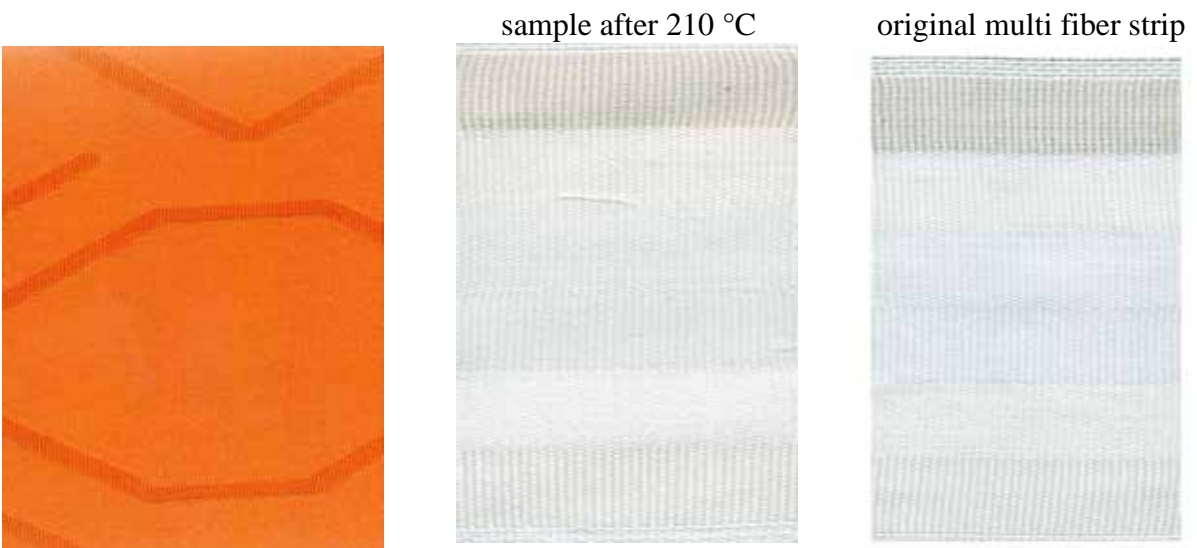


Figure 214: aNIR[®] Dianix[®] Yellow Brown XF2 medium shade (Setup 2) sample after 210 °C

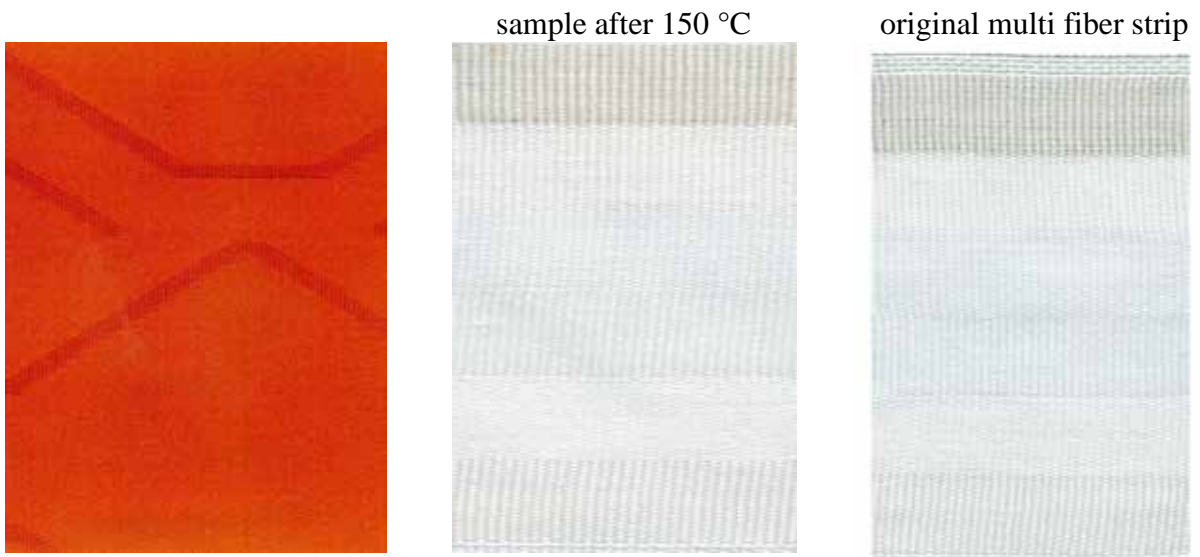


Figure 215: aNIR[®] Dianix[®] Yellow Brown XF2 dark shade (Setup 2) sample after 150 °C

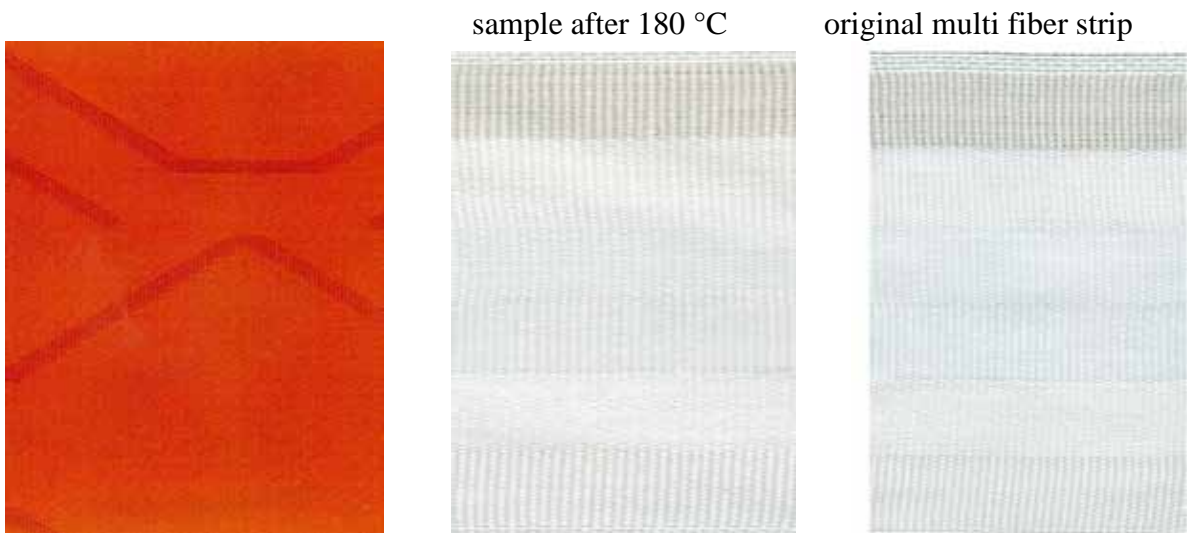


Figure 216: aNIR[®] Dianix[®] Yellow Brown XF2 dark shade (Setup 2) sample after 180 °C



Figure 217: aNIR[®] Dianix[®] Yellow Brown XF2 dark shade (Setup 2) sample after 210 °C



Figure 218: aNIR[®] Dianix[®] Yellow Brown CC pale shade (Setup 2) sample after 150 °C

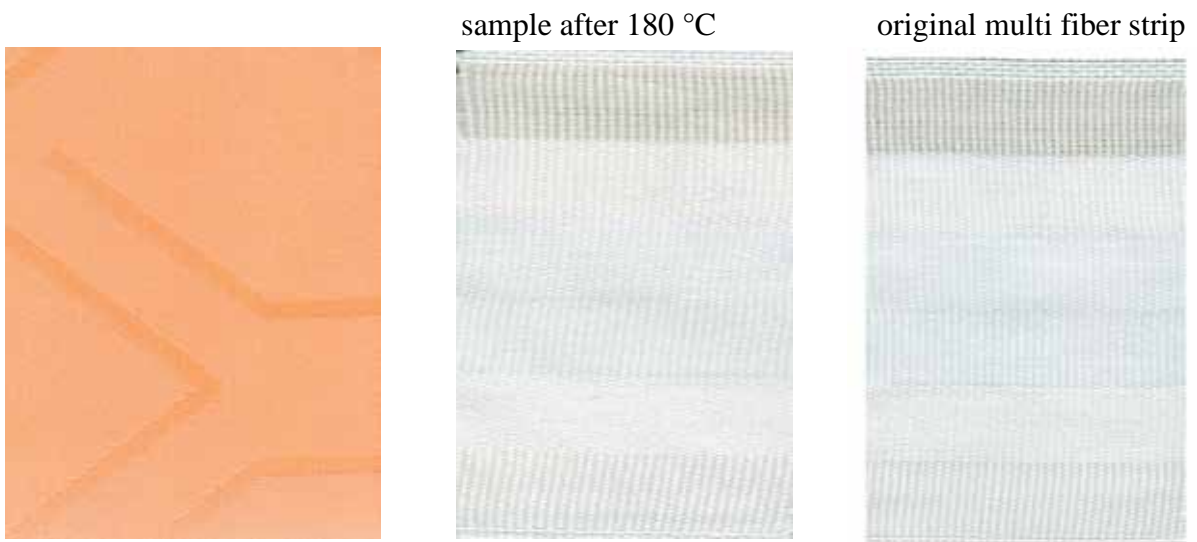


Figure 219: aNIR[®] Dianix[®] Yellow Brown CC pale shade (Setup 2) sample after 180 °C



Figure 220: aNIR[®] Dianix[®] Yellow Brown CC pale shade (Setup 2) sample after 210 °C



Figure 221: aNIR® Dianix® Yellow Brown CC medium shade (Setup 2) sample after 150 °C

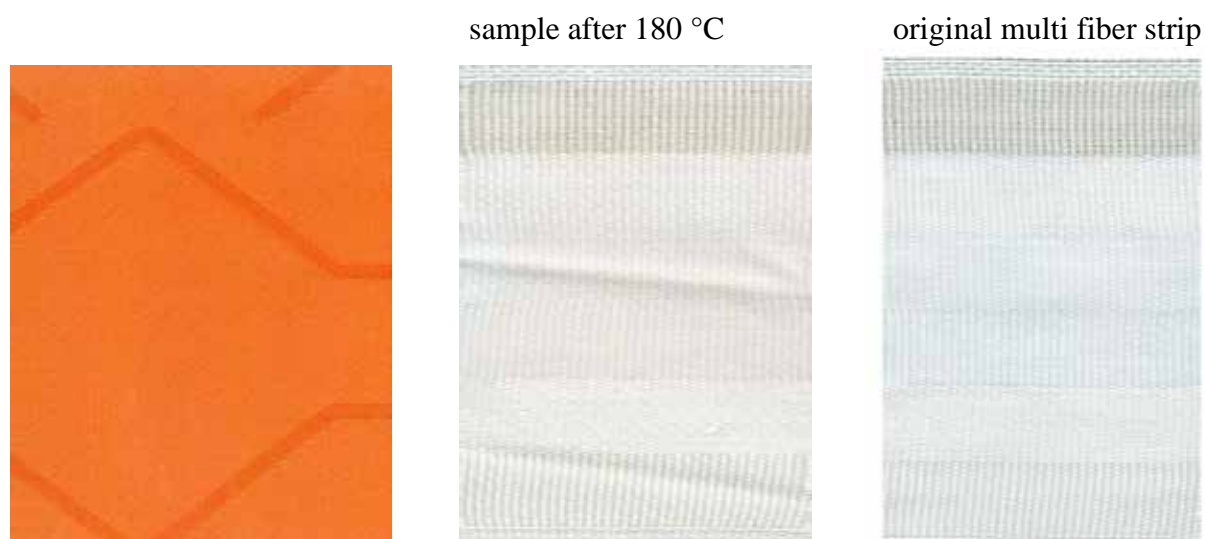


Figure 222: aNIR® Dianix® Yellow Brown CC medium shade (Setup 2) sample after 180 °C



Figure 223: aNIR® Dianix® Yellow Brown CC medium shade (Setup 2) sample after 210 °C

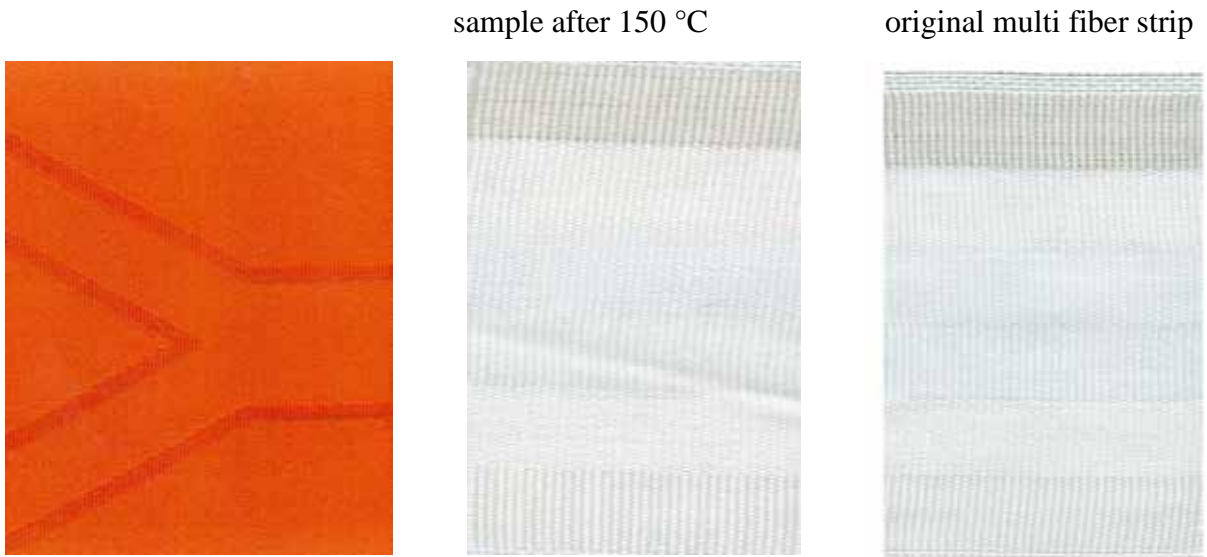


Figure 224: aNIR[®] Dianix[®] Yellow Brown CC dark shade (Setup 2) sample after 150 °C

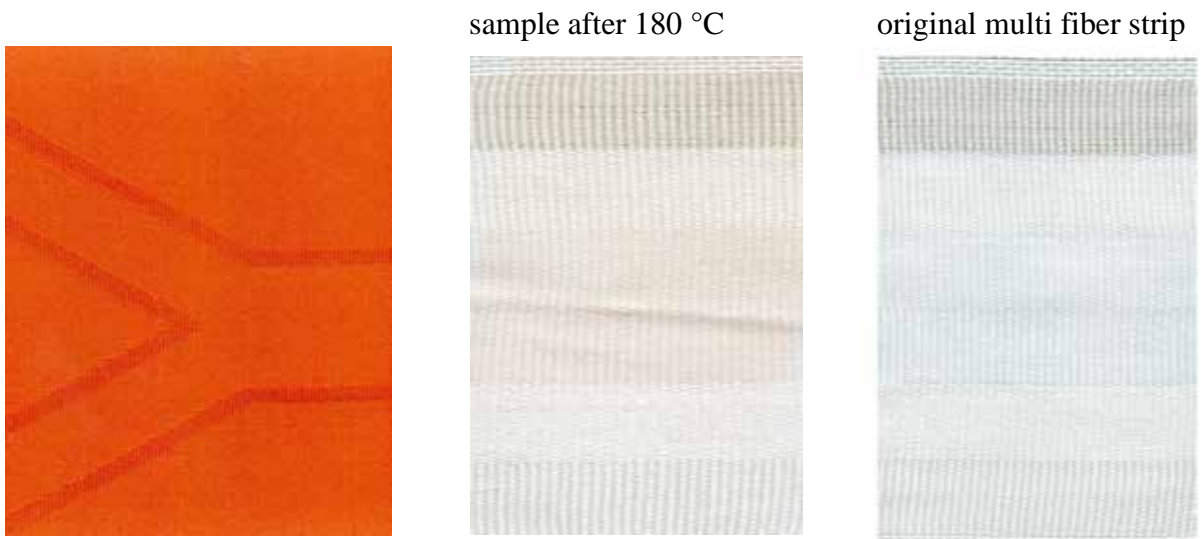


Figure 225: aNIR[®] Dianix[®] Yellow Brown CC dark shade (Setup 2) sample after 180 °C



Figure 226: aNIR[®] Dianix[®] Yellow Brown CC dark shade (Setup 2) sample after 210 °C

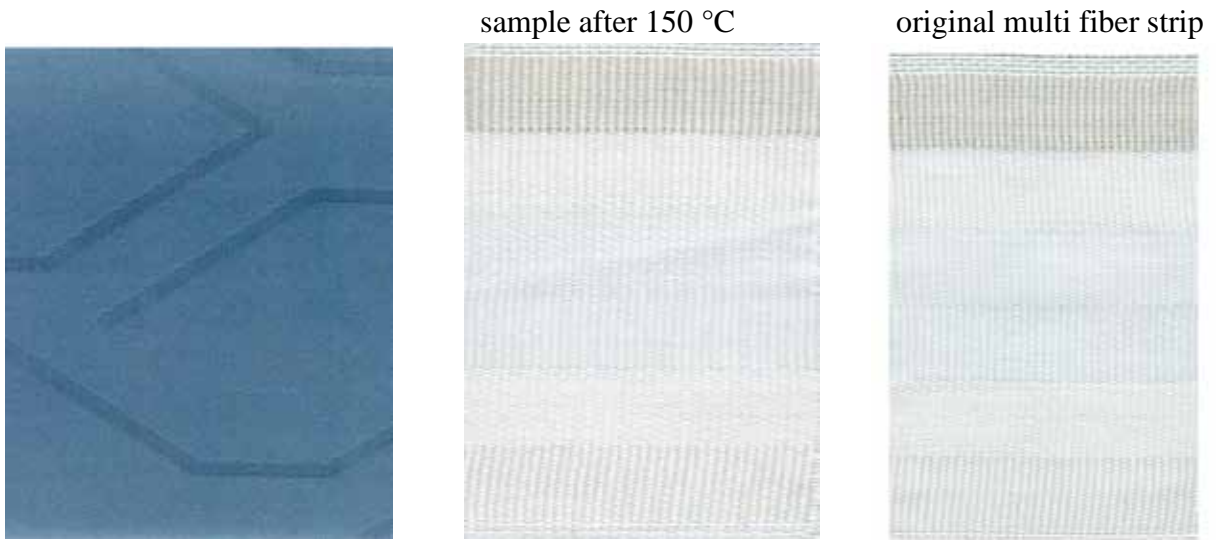


Figure 227: convection process Dianix® Navy XF2 pale shade sample after 150 °C

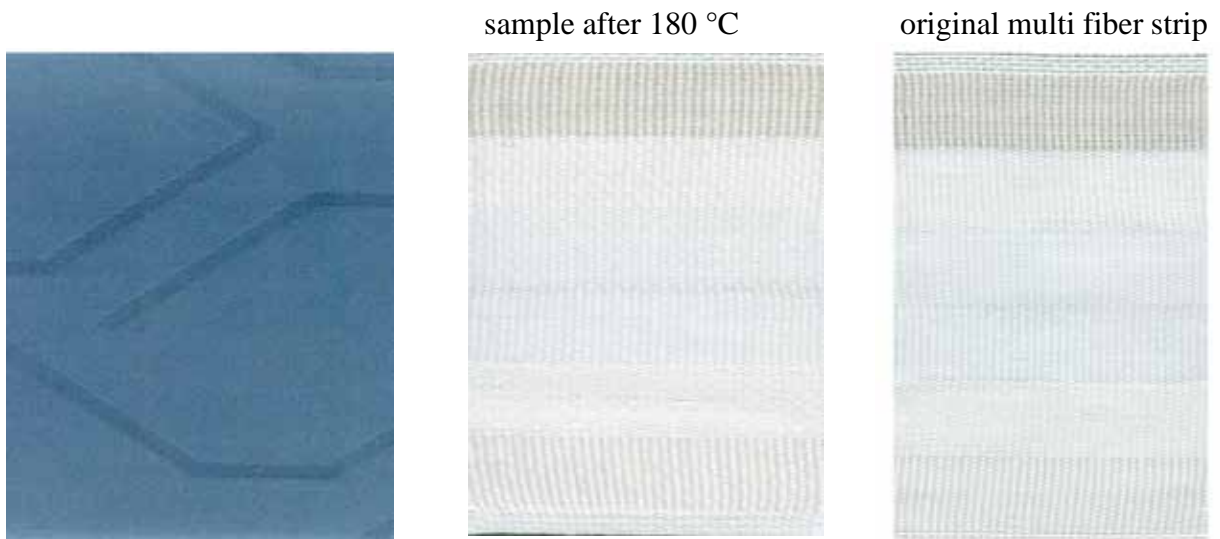


Figure 228: convection process Dianix® Navy XF2 pale shade sample after 180 °C

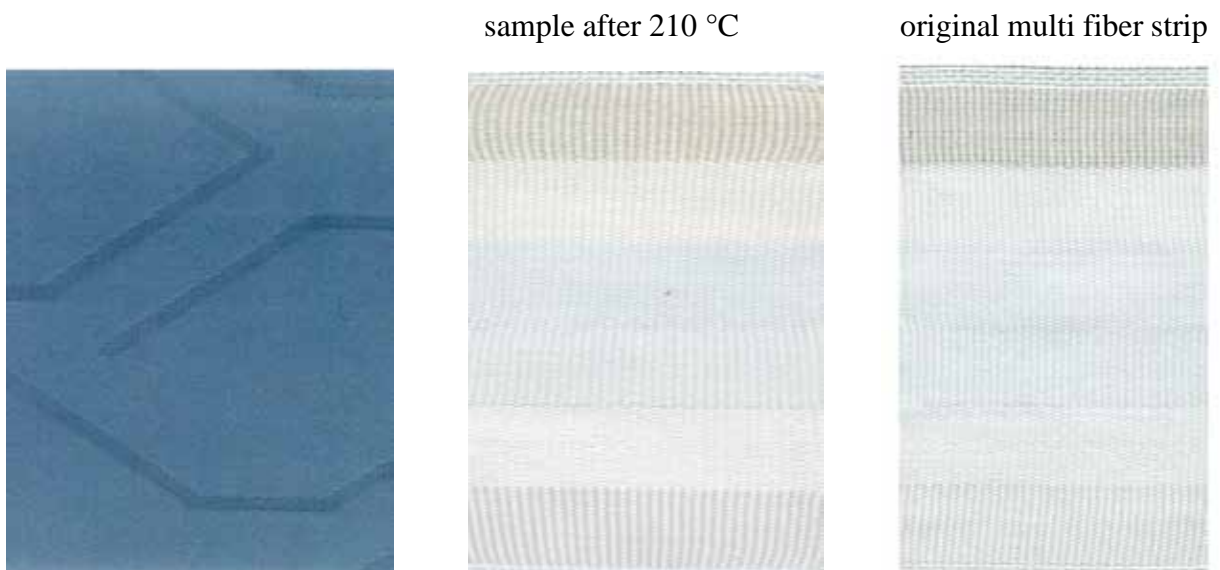


Figure 229: convection process Dianix® Navy XF2 pale shade sample after 210 °C

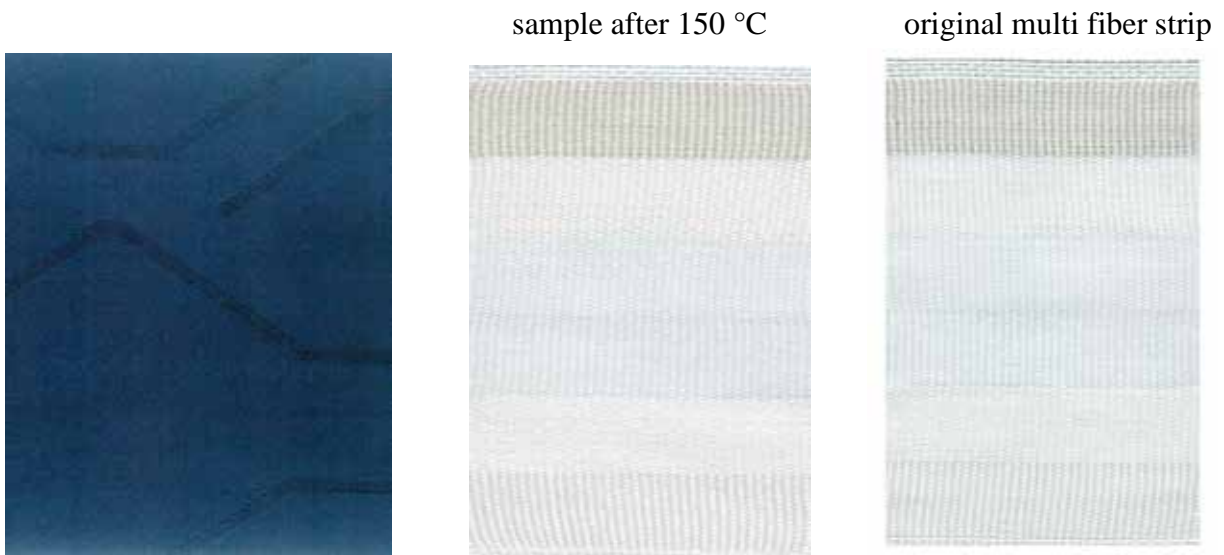


Figure 230: convection process Dianix® Navy XF2 medium shade sample after 150 °C

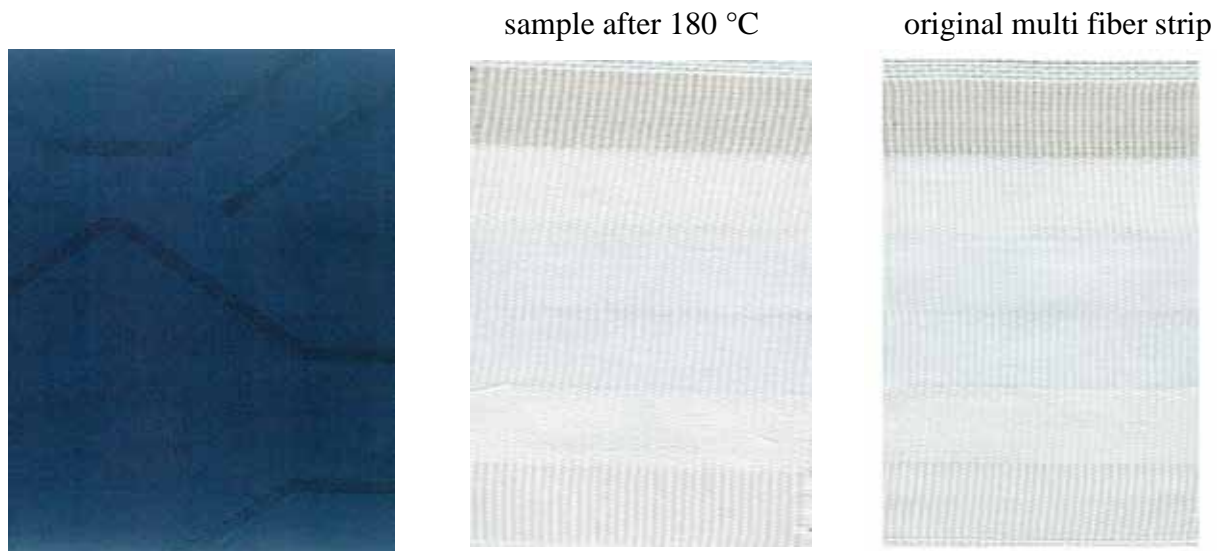


Figure 231: convection process Dianix® Navy XF2 medium shade sample after 180 °C

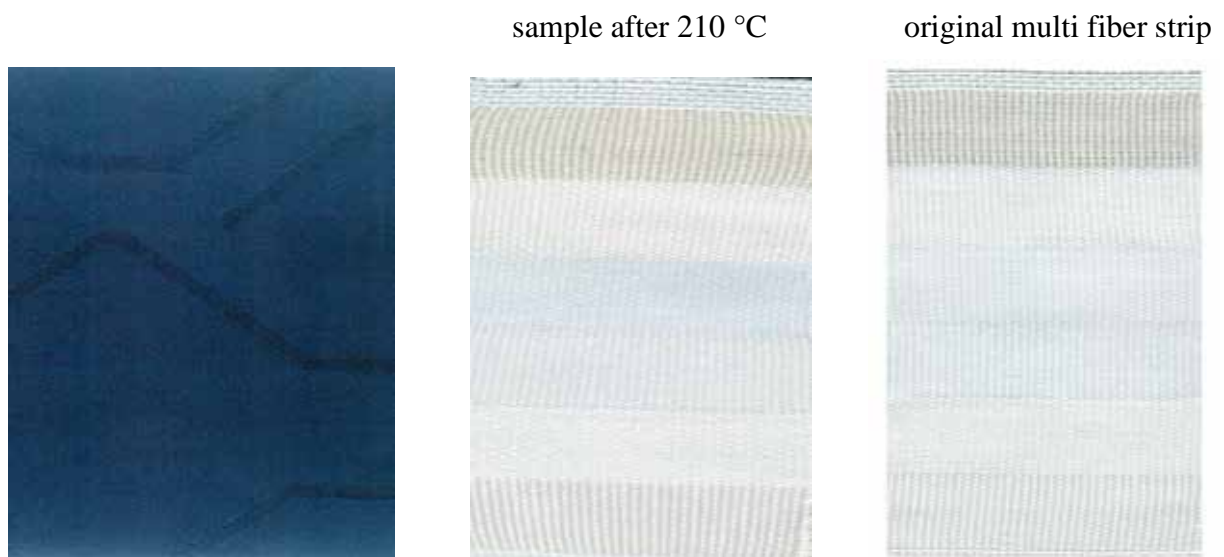


Figure 232: convection process Dianix® Navy XF2 medium shade sample after 210 °C



Figure 233: convection process Dianix® Navy XF2 dark shade sample after 150 °C



Figure 234: convection process Dianix® Navy XF2 dark shade sample after 180 °C

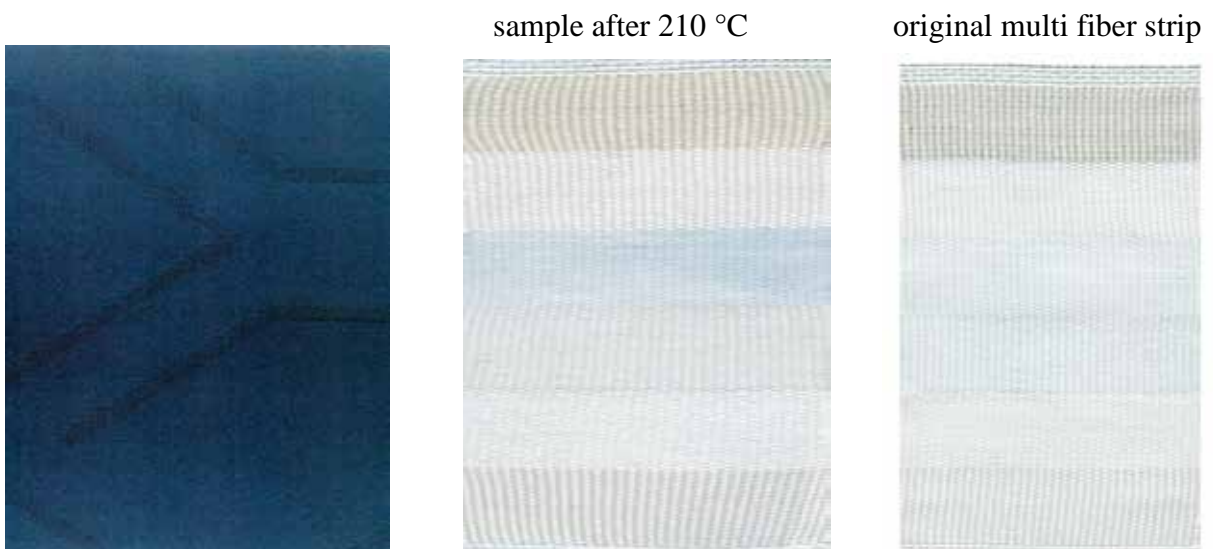


Figure 235: convection process Dianix® Navy XF2 dark shade sample after 210 °C

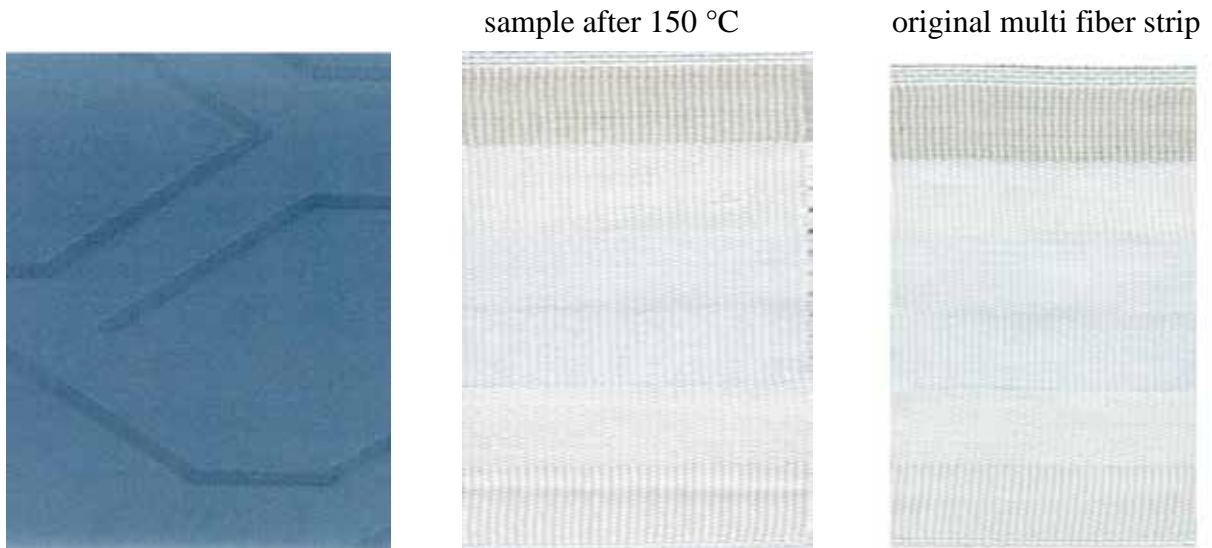


Figure 236: aNIR® Dianix® Navy XF2 pale shade (setup 1) sample after 150 °C

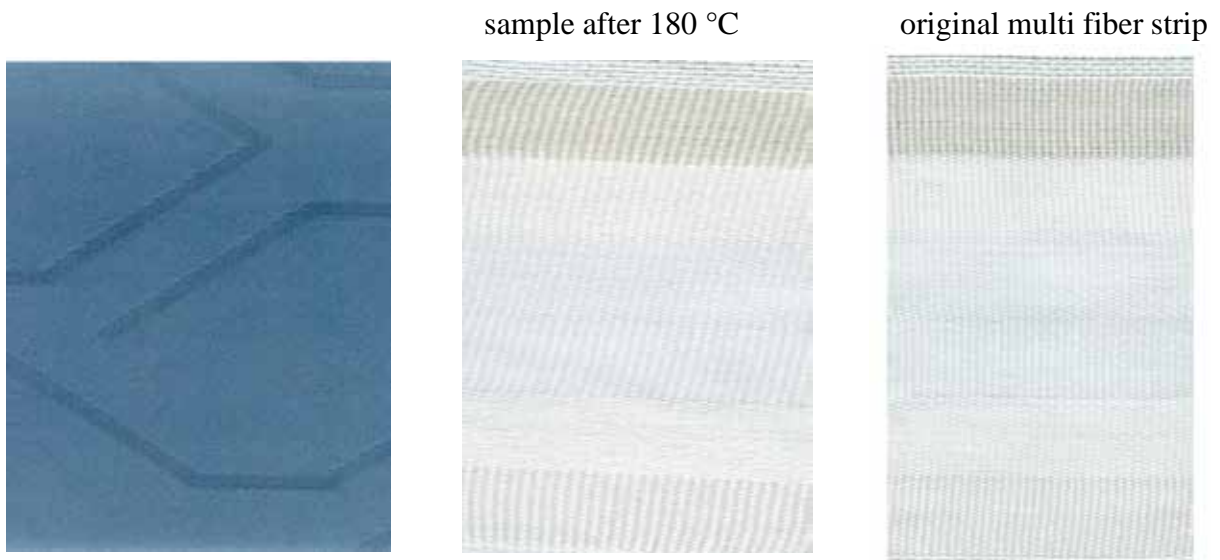


Figure 237: aNIR® Dianix® Navy XF2 pale shade (setup 1) sample after 180 °C

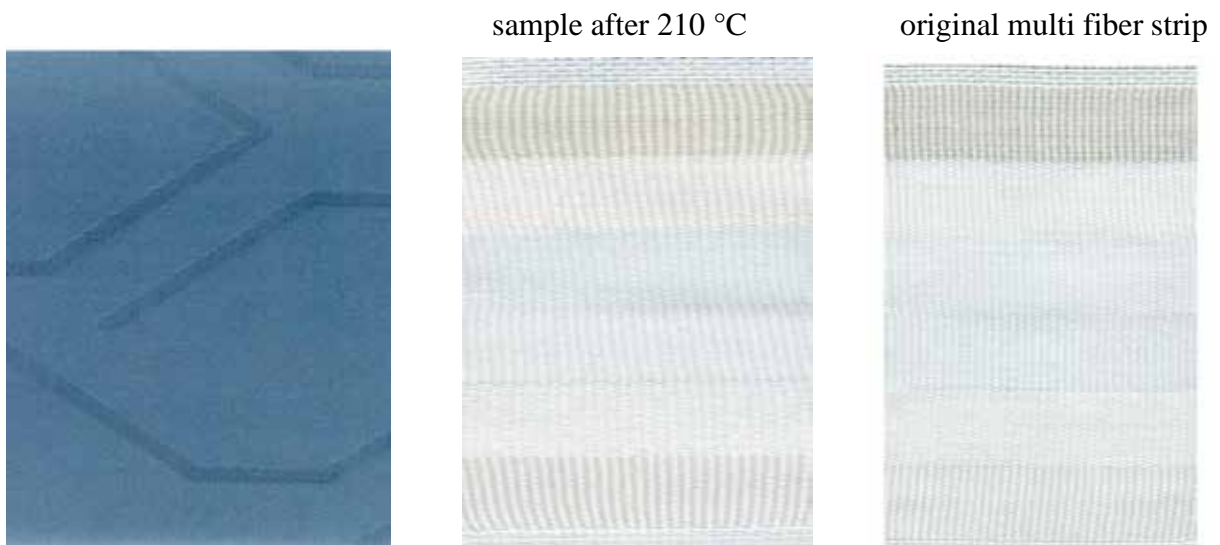


Figure 238: aNIR® Dianix® Navy XF2 pale shade (setup 1) sample after 210 °C



Figure 239: aNIR® Dianix® Navy XF2 medium shade (setup 1) sample after 150 °C

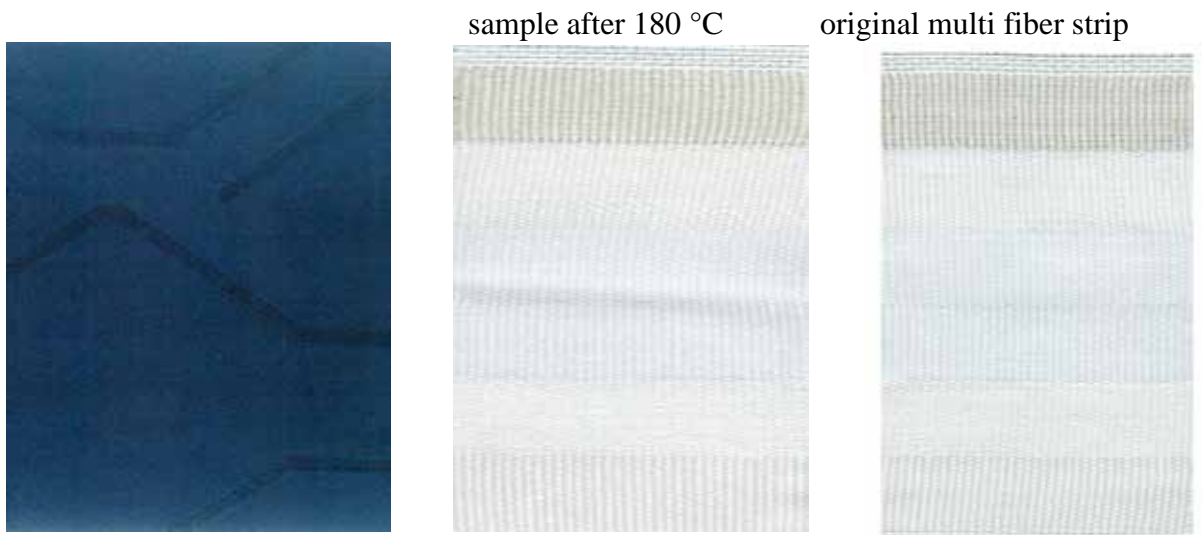


Figure 240: aNIR® Dianix® Navy XF2 medium shade (setup 1) sample after 180 °C

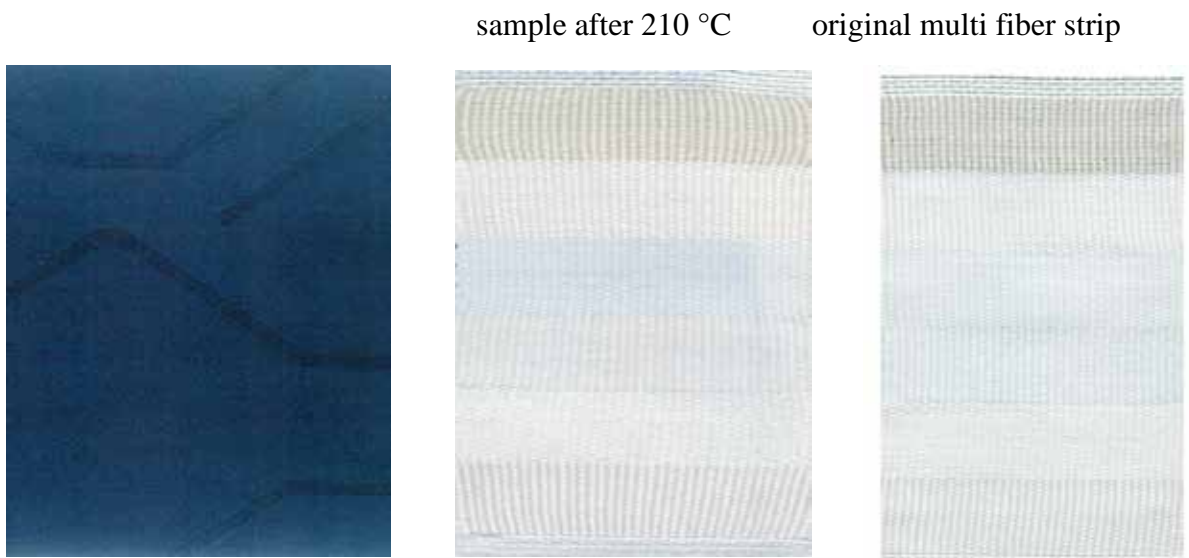


Figure 241: aNIR® Dianix® Navy XF2 medium shade (setup 1) sample after 210 °C

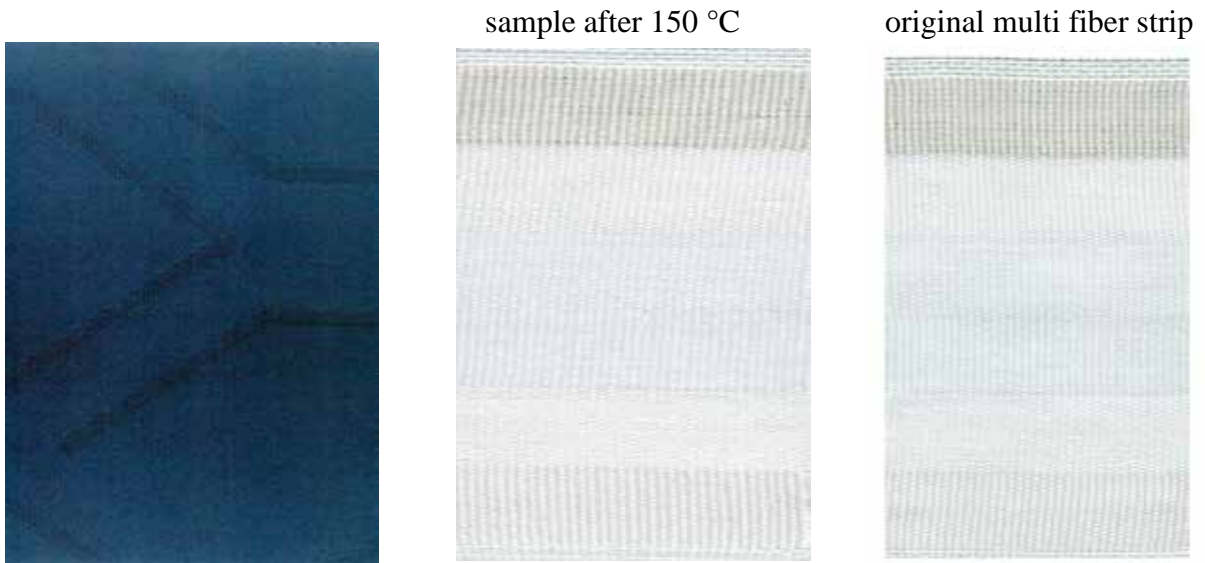


Figure 242: aNIR® Dianix® Navy XF2 dark shade (setup 1) sample after 150 °C



Figure 243: aNIR® Dianix® Navy XF2 dark shade (setup 1) sample after 180 °C



Figure 244: aNIR® Dianix® Navy XF2 dark shade (setup 1) sample after 210 °C



Figure 245: aNIR® Dianix® Navy XF2 pale shade (setup 2) sample after 150 °C

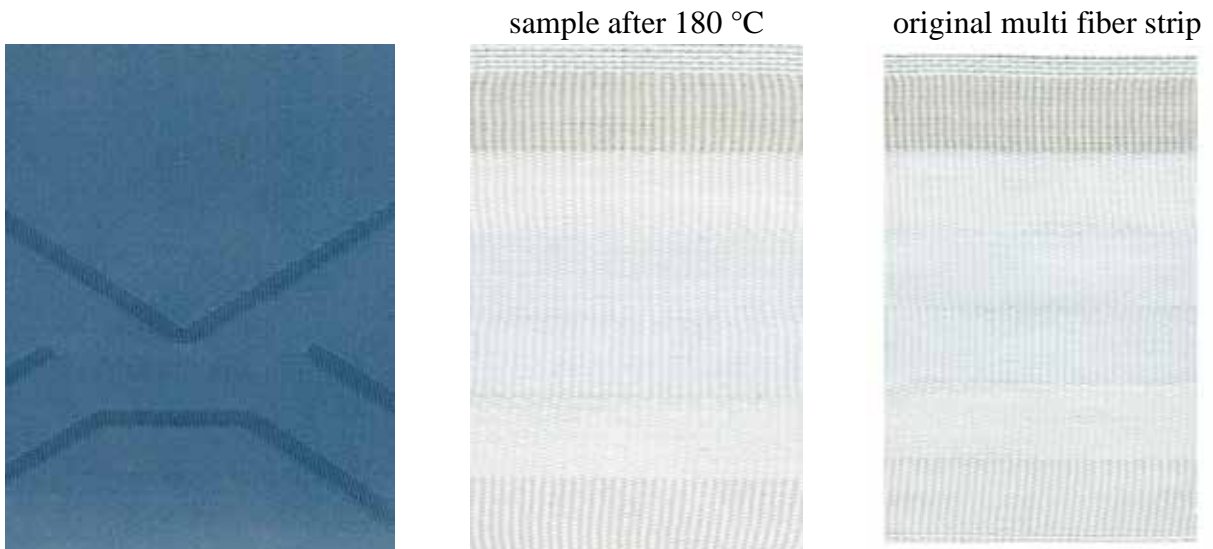


Figure 246: aNIR® Dianix® Navy XF2 pale shade (setup 2) sample after 180 °C

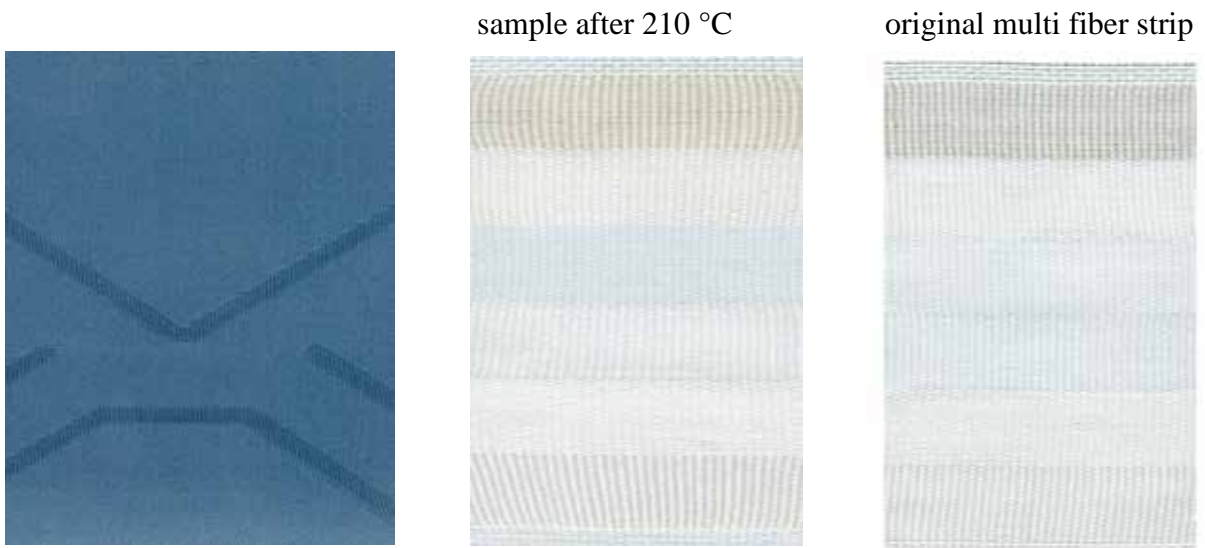


Figure 247: aNIR® Dianix® Navy XF2 pale shade (setup 2) sample after 210 °C



Figure 248: aNIR® Dianix® Navy XF2 medium shade (setup 2) sample after 150 °C



Figure 249: aNIR® Dianix® Navy XF2 medium shade (setup 2) sample after 180 °C

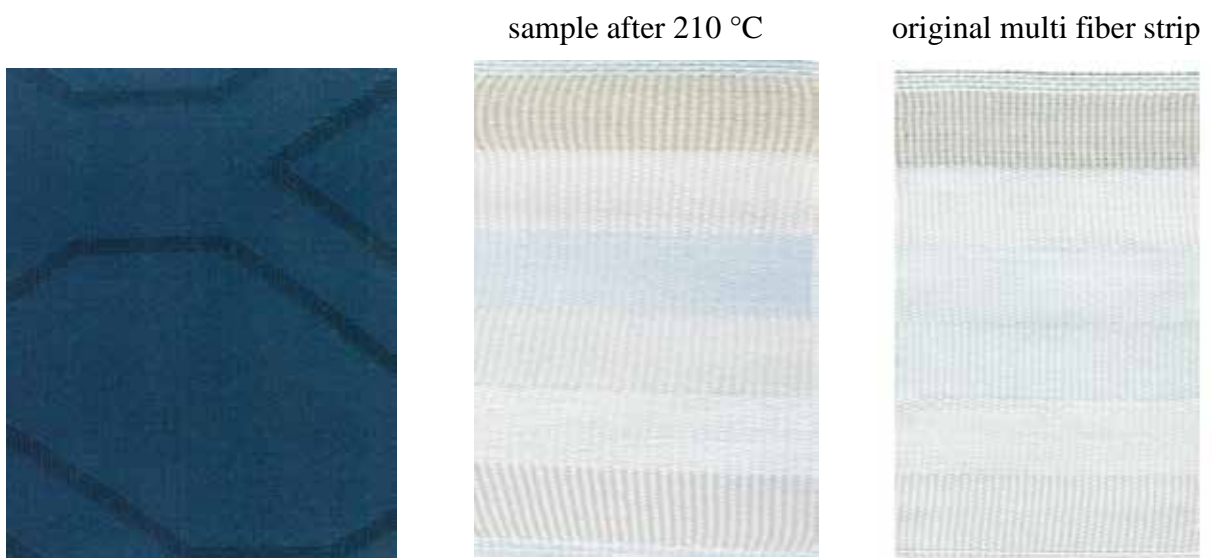


Figure 250: aNIR® Dianix® Navy XF2 medium shade (setup 2) sample after 210 °C

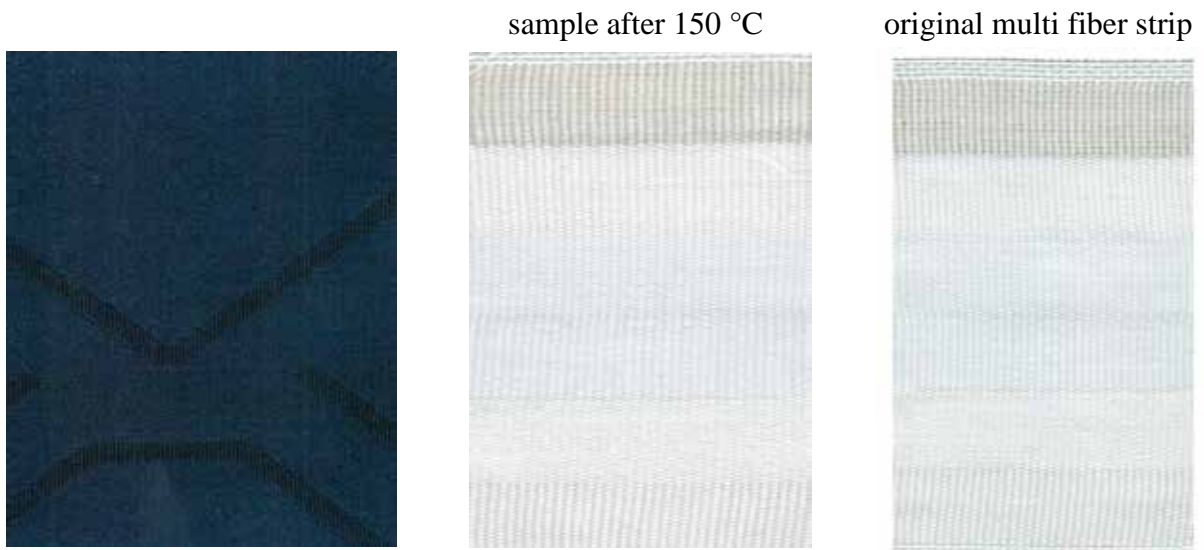


Figure 251: aNIR® Dianix® Navy XF2 dark shade (setup 2) sample after 150 °C



Figure 252: aNIR® Dianix® Navy XF2 dark shade (setup 2) sample after 180 °C



Figure 253: aNIR® Dianix® Navy XF2 dark shade (setup 2) sample after 210 °C

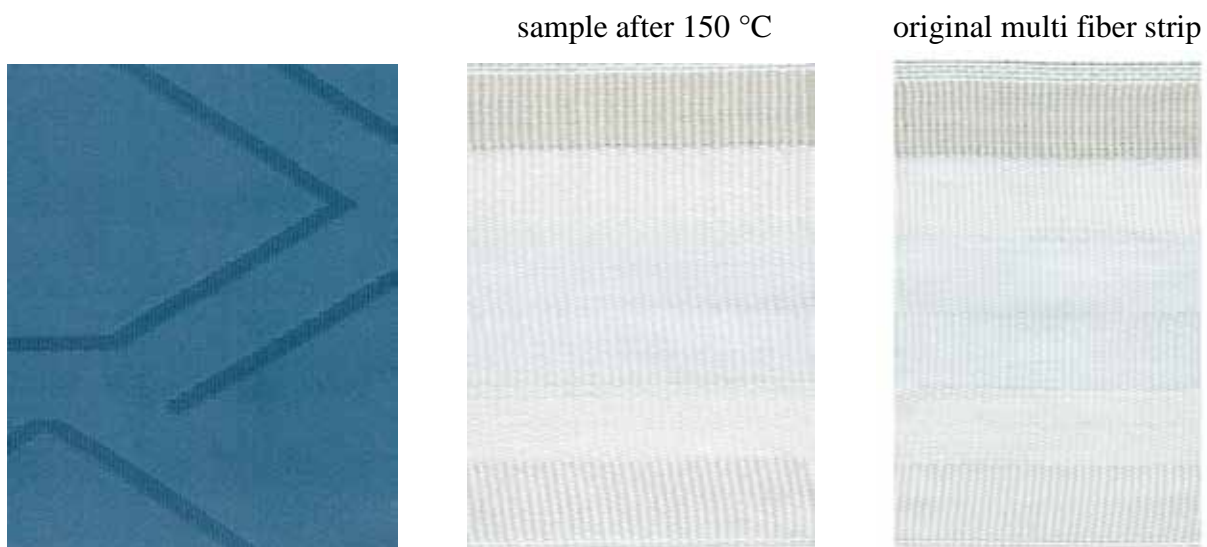


Figure 254: aNIR® Dianix® Navy CC pale shade (setup 2) sample after 180 °C

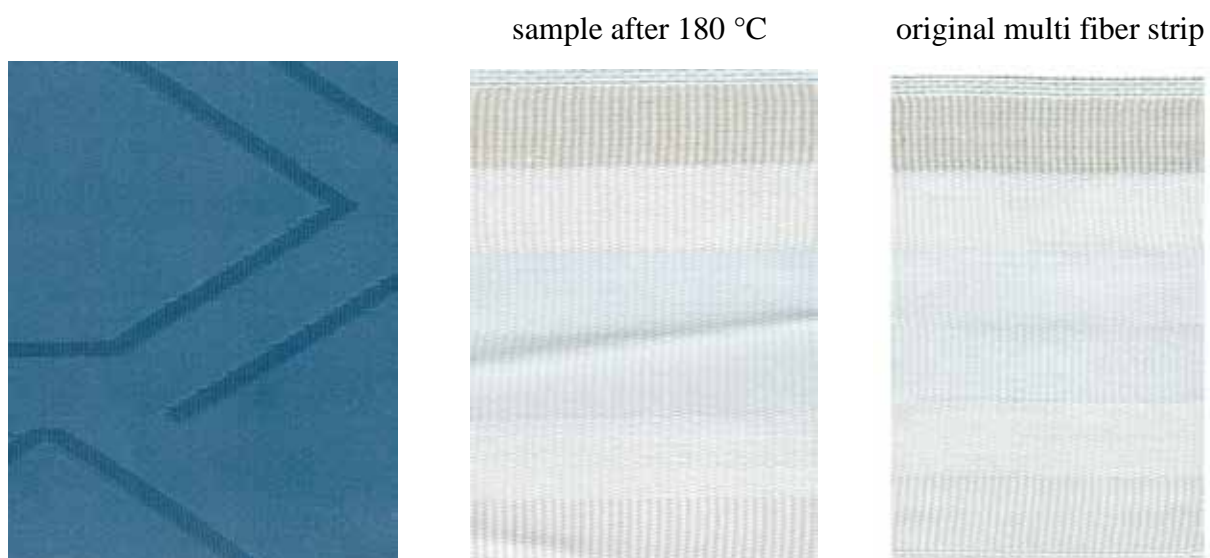


Figure 255: aNIR® Dianix® Navy CC pale shade (setup 2) sample after 180 °C

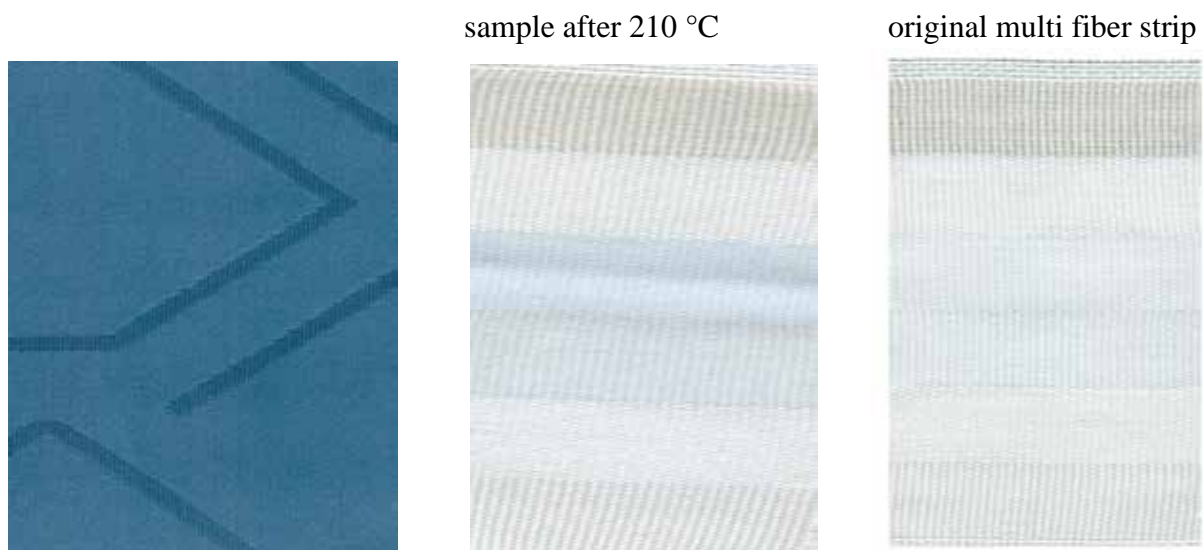


Figure 256: aNIR® Dianix® Navy CC pale shade (setup 2) sample after 210 °C



Figure 257: aNIR® Dianix® Navy CC medium shade (setup 2) sample after 150 °C

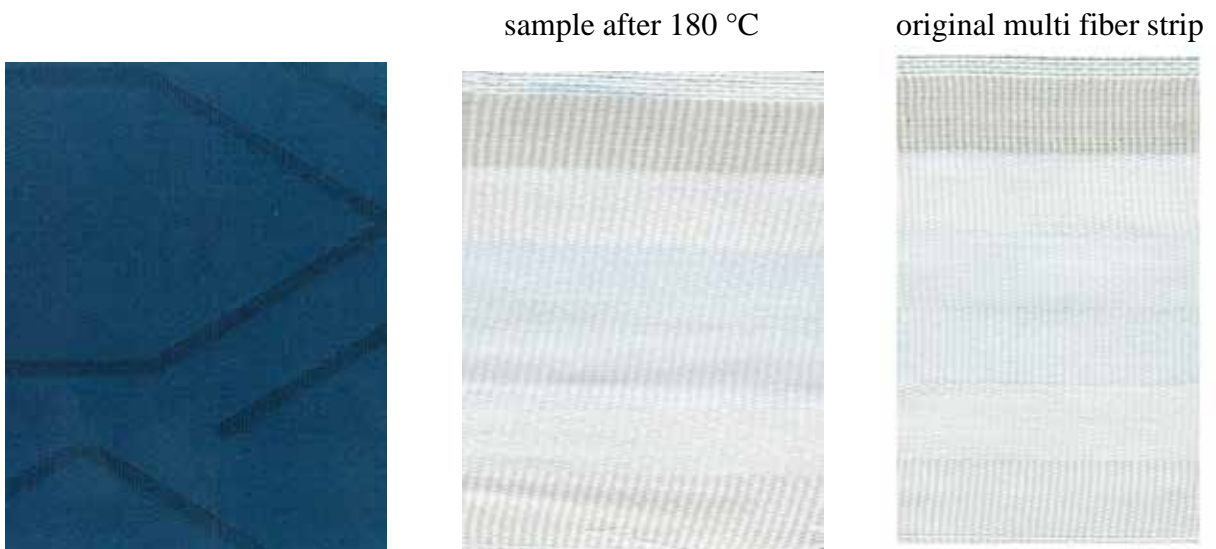


Figure 258: aNIR® Dianix® Navy CC medium shade (setup 2) sample after 180 °C

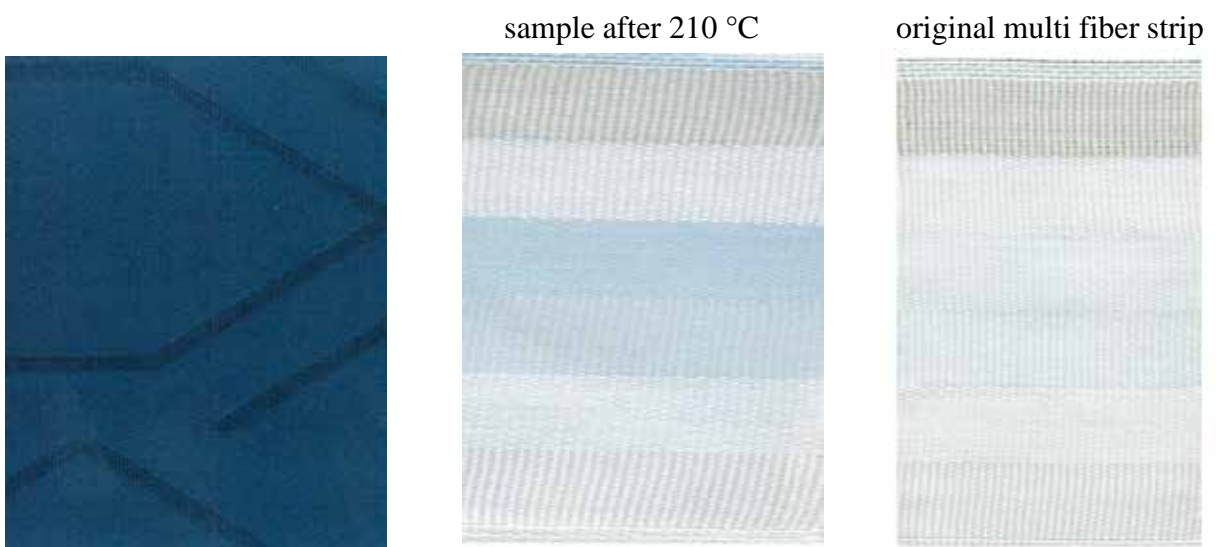


Figure 259: aNIR® Dianix® Navy CC medium shade (setup 2) sample after 210 °C

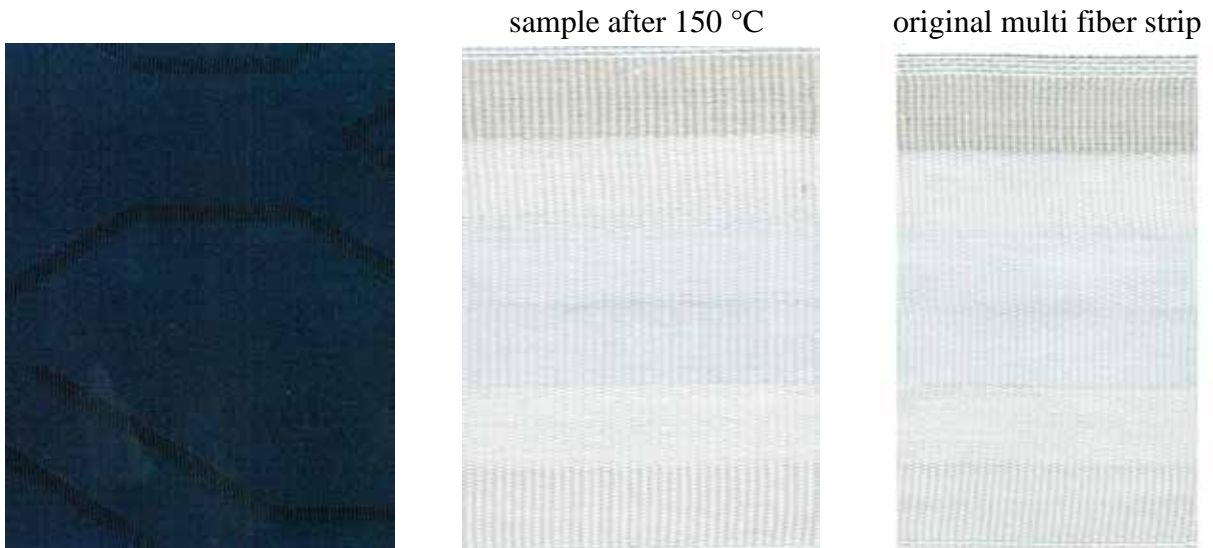


Figure 260: aNIR[®] Dianix[®] Navy CC dark shade (setup 2) sample after 210 °C



Figure 261: aNIR[®] Dianix[®] Navy CC dark shade (setup 2) sample after 180 °C

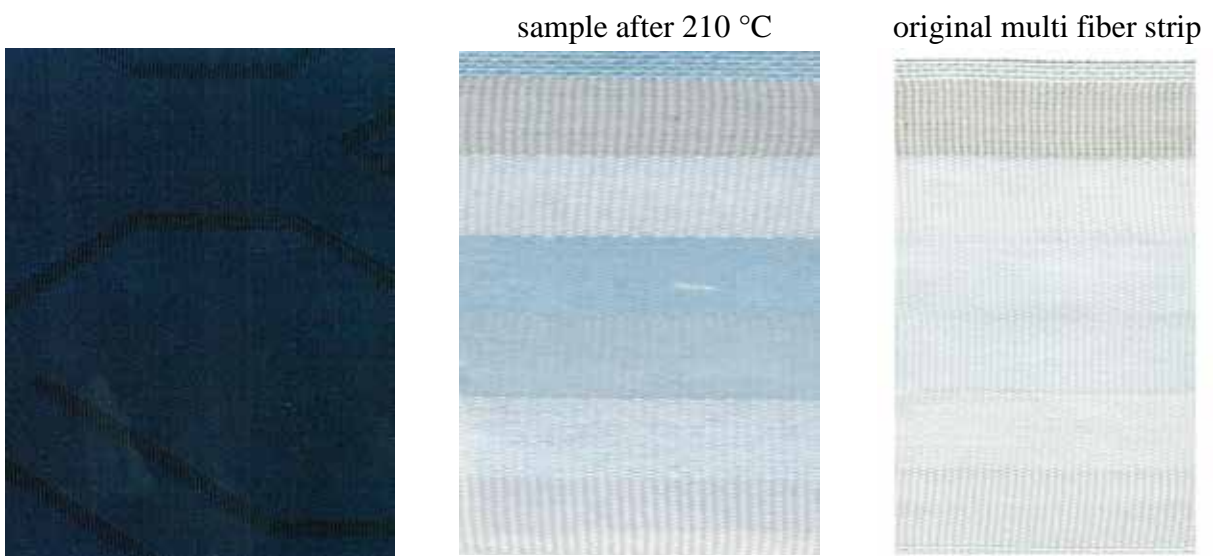


Figure 262: aNIR[®] Dianix[®] Navy CC dark shade (setup 2) sample after 210 °C

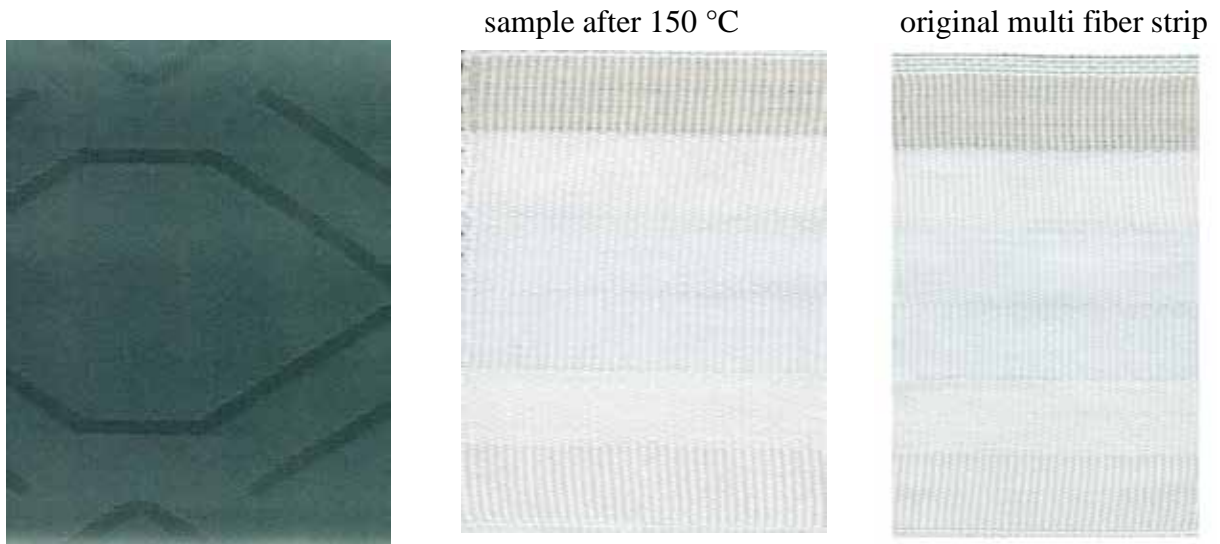


Figure 263: convection process Dianix® Black XF2 300% pale shade sample after 150 °C

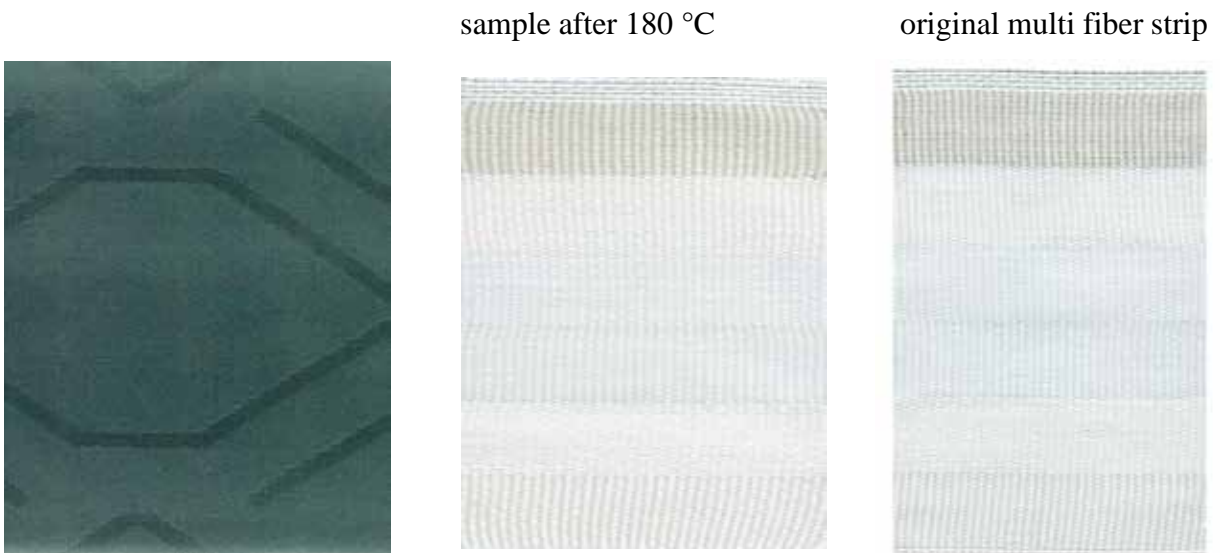


Figure 264: convection process Dianix® Black XF2 300% pale shade sample after 180 °C

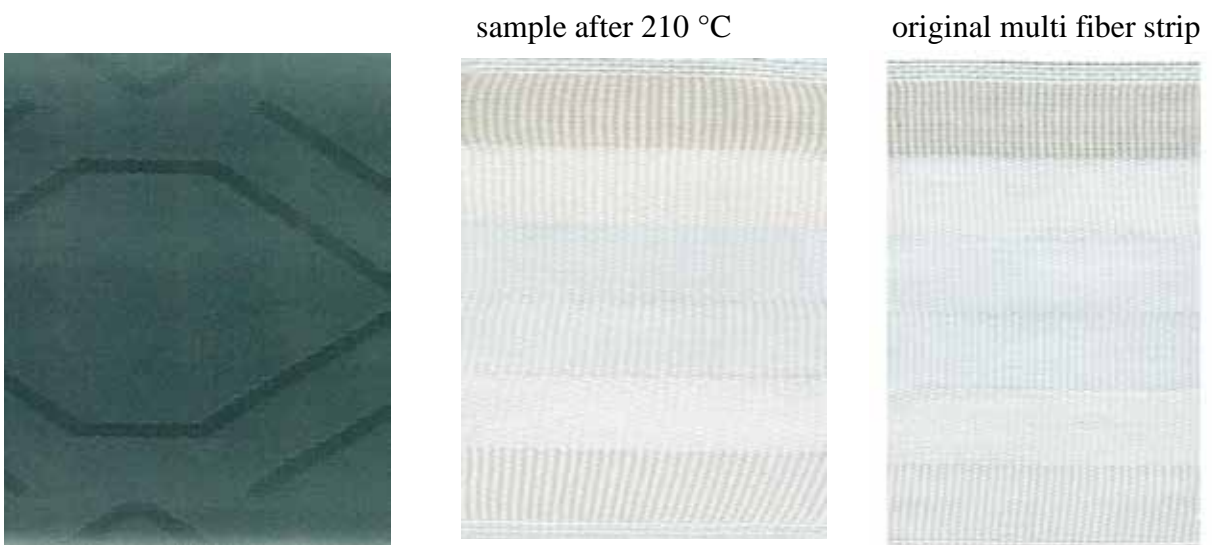


Figure 265: convection process Dianix® Black XF2 300% pale shade sample after 210 °C

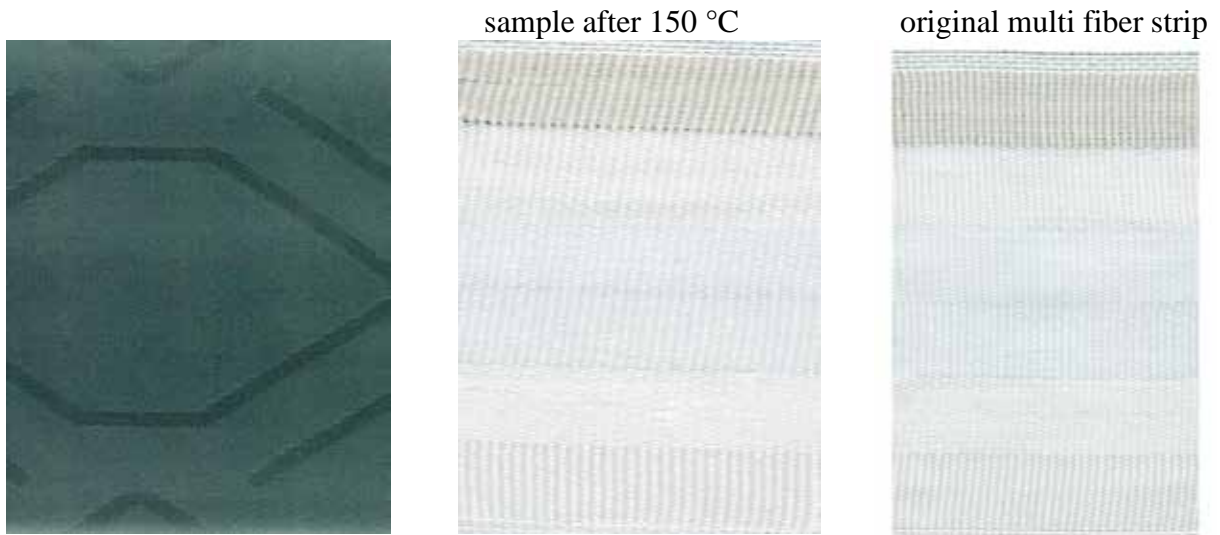


Figure 266: convection process Dianix® Black XF2 300% medium shade sample after 150 °C

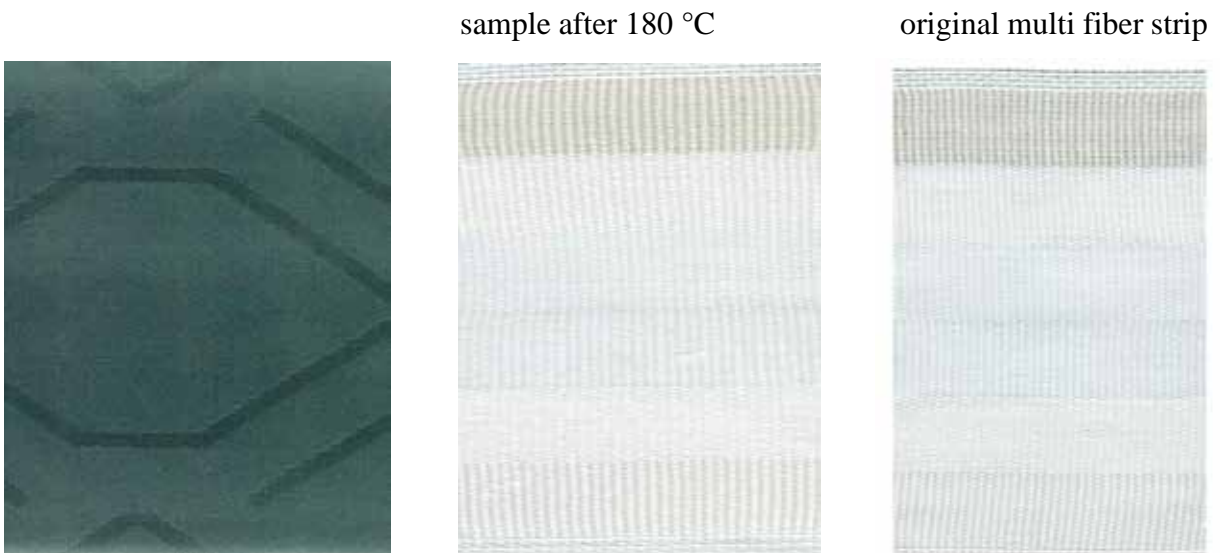


Figure 267: convection process Dianix® Black XF2 300% medium shade sample after 180 °C

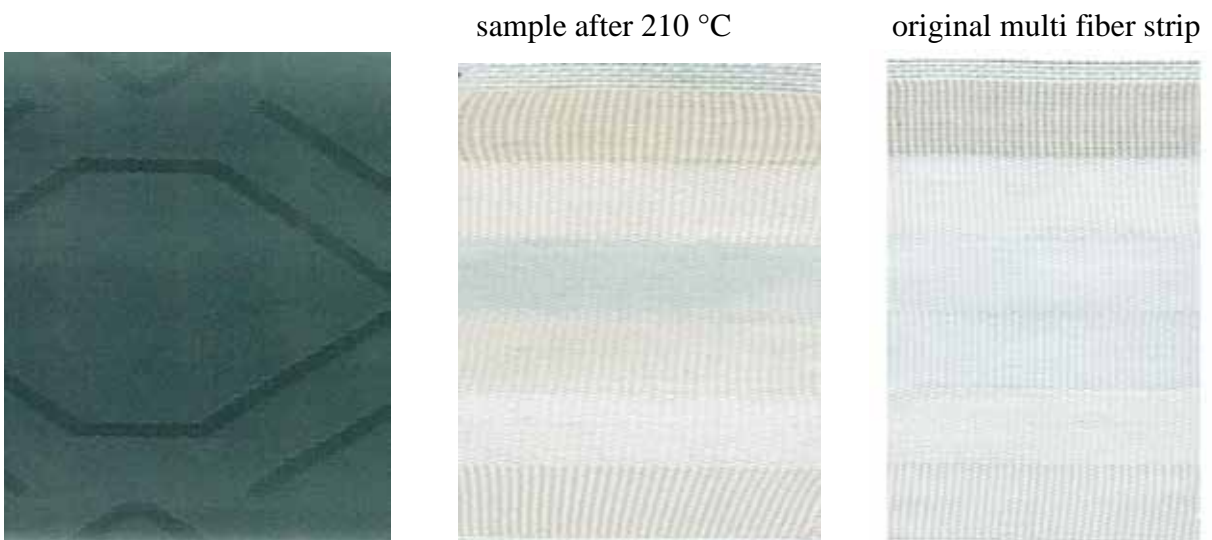


Figure 268: convection process Dianix® Black XF2 300% medium shade sample after 210 °C

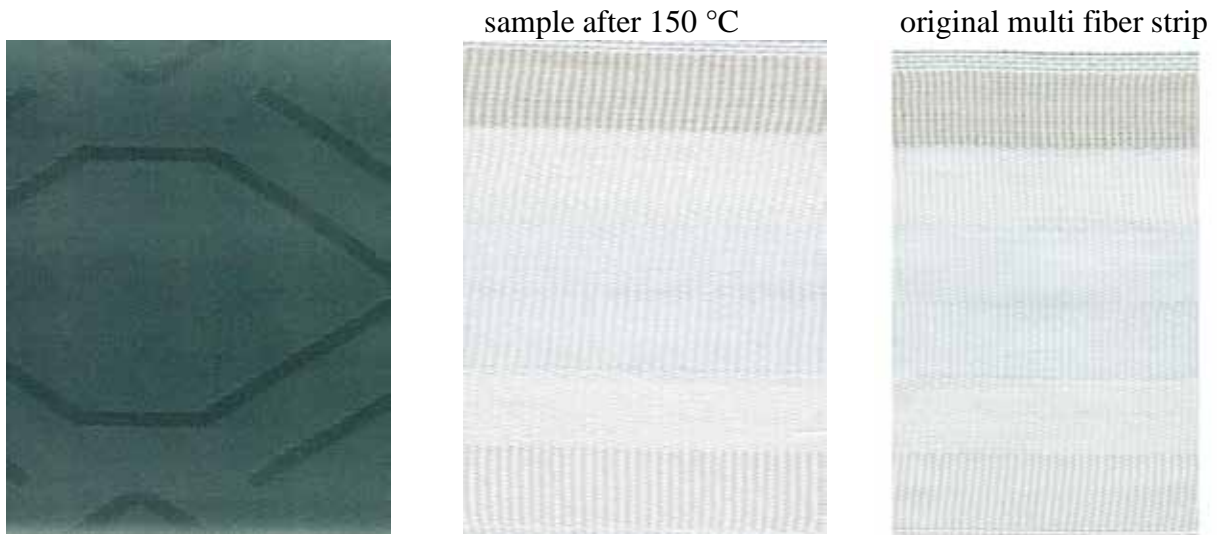


Figure 269: convection process Dianix® Black XF2 300% dark shade sample after 150 °C

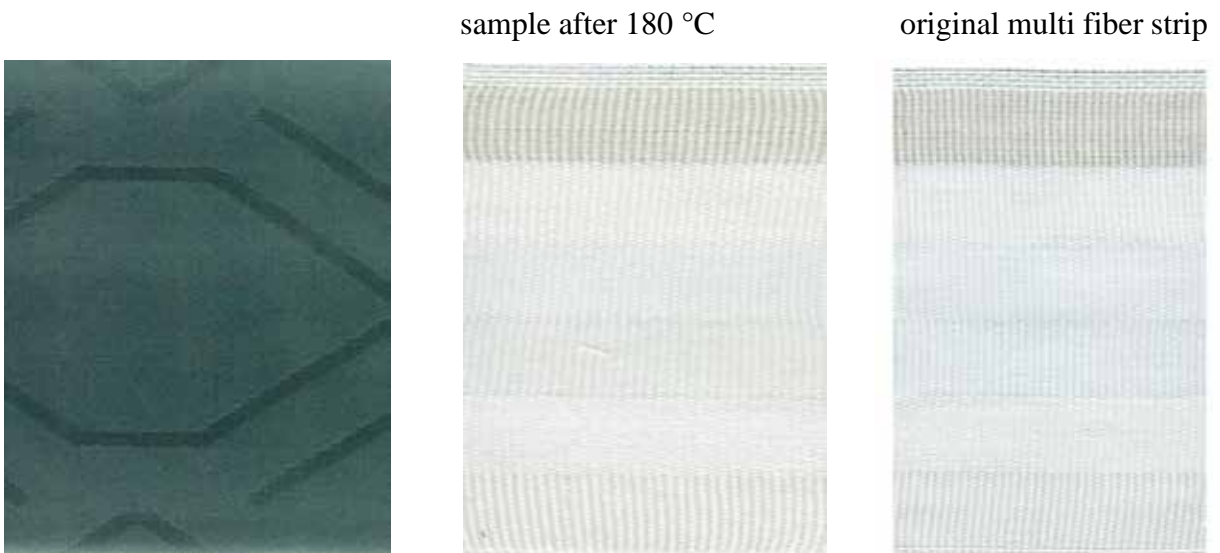


Figure 270: convection process Dianix® Black XF2 300% dark shade sample after 180 °C

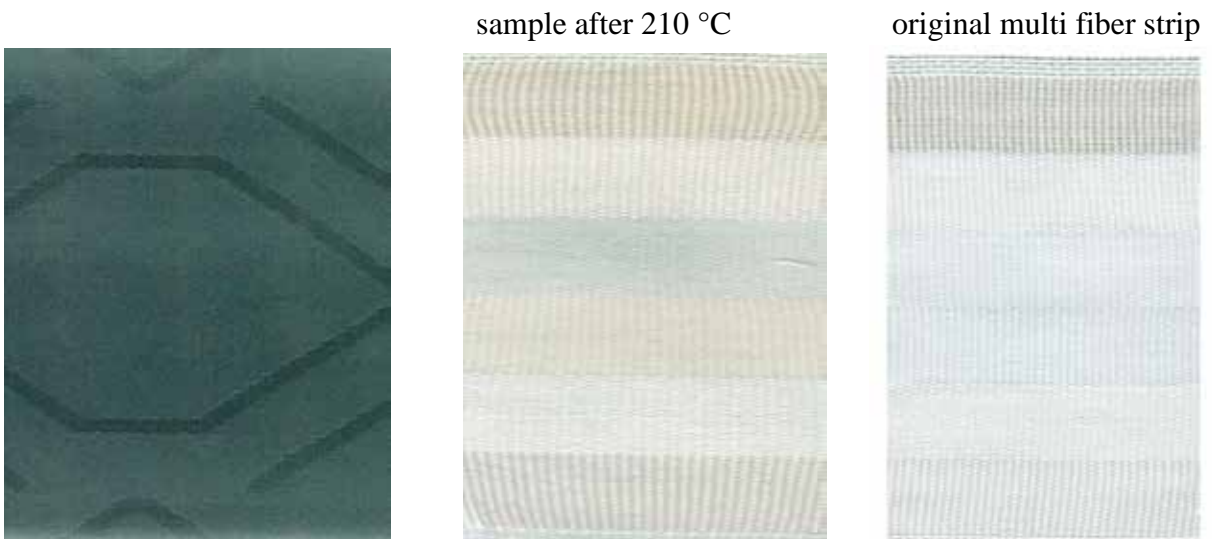


Figure 271: convection process Dianix® Black XF2 300% dark shade sample after 210 °C

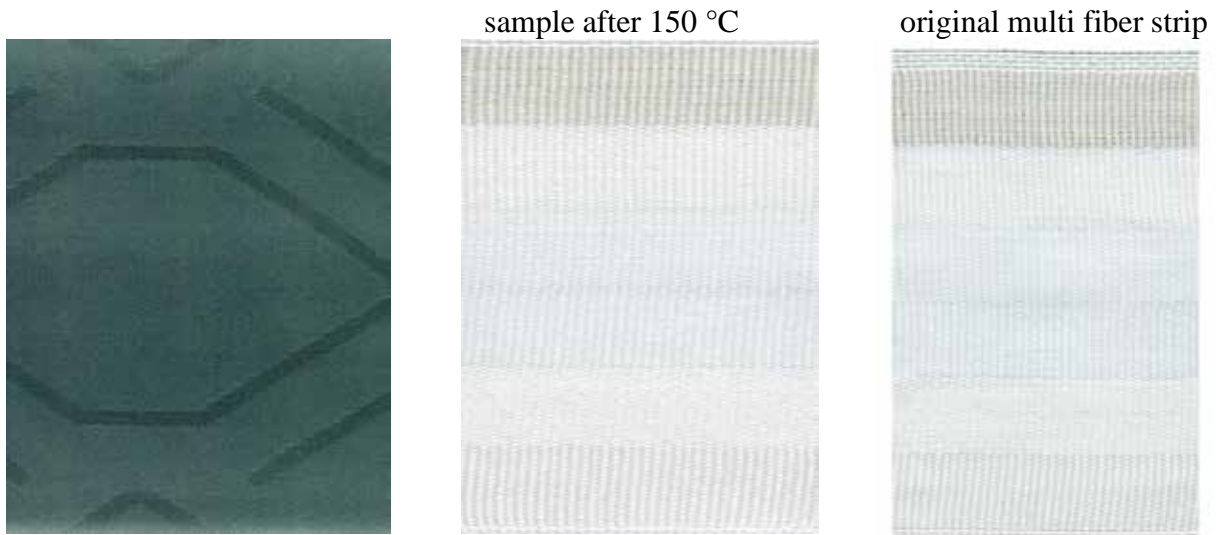


Figure 272: aNIR® Dianix® Black XF2 300% pale shade (setup 1) sample after 150 °C

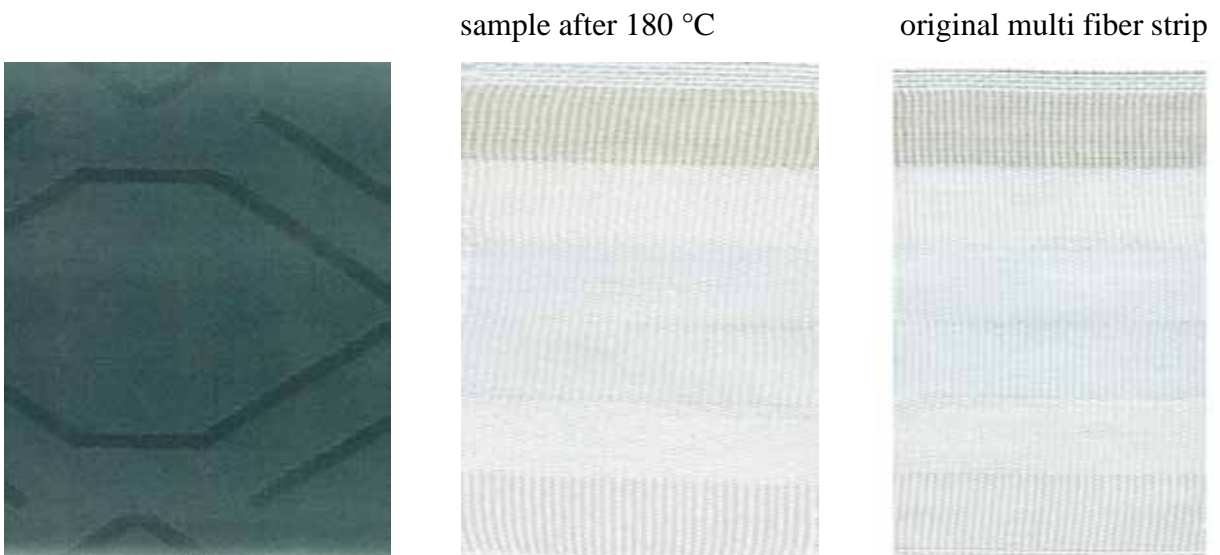


Figure 273: aNIR® Dianix® Black XF2 300% pale shade (setup 1) sample after 180 °C

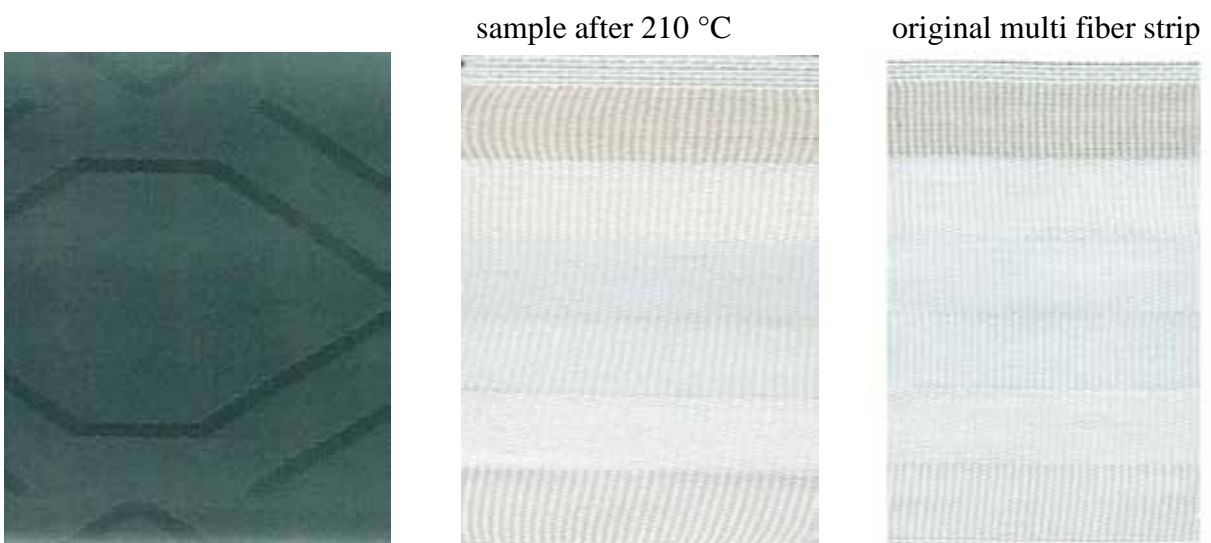


Figure 274: aNIR® Dianix® Black XF2 300% pale shade (setup 1) sample after 210 °C



Figure 275: aNIR[®] Dianix[®] Black XF2 300% medium shade (setup 1) sample after 150 °C

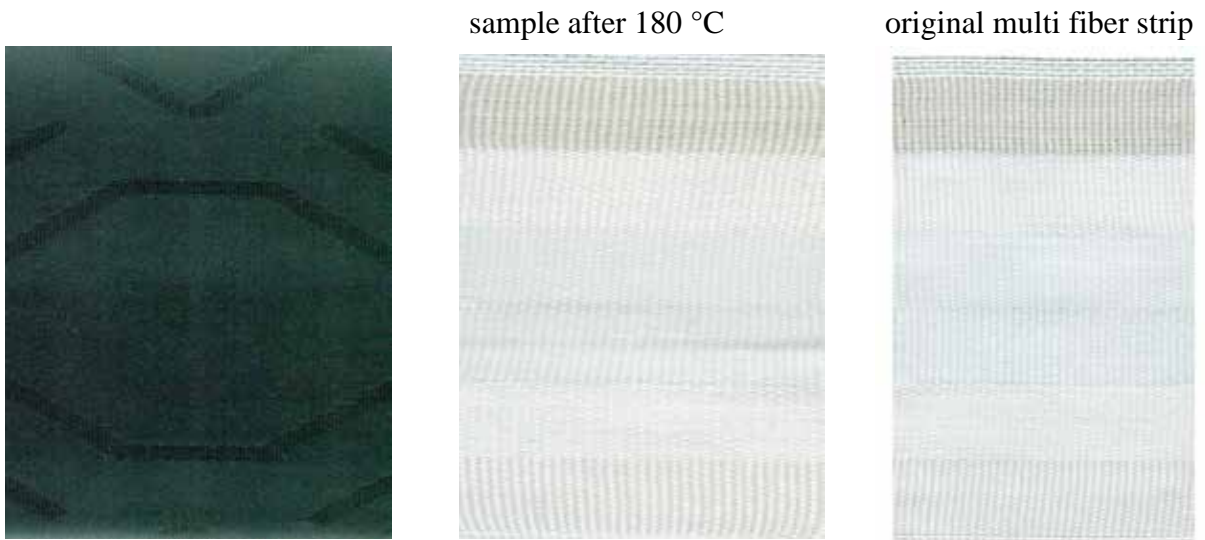


Figure 276: aNIR[®] Dianix[®] Black XF2 300% medium shade (setup 1) sample after 180 °C



Figure 277: aNIR[®] Dianix[®] Black XF2 300% medium shade (setup 1) sample after 210 °C

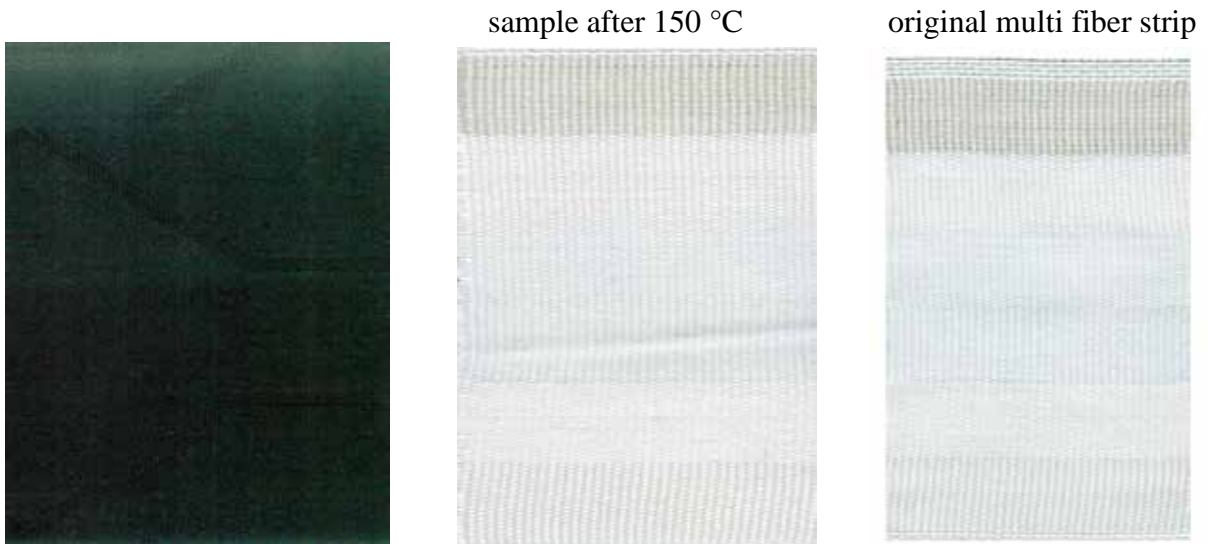


Figure 278: aNIR® Dianix® Black XF2 300% dark shade (setup 1) sample after 210 °C

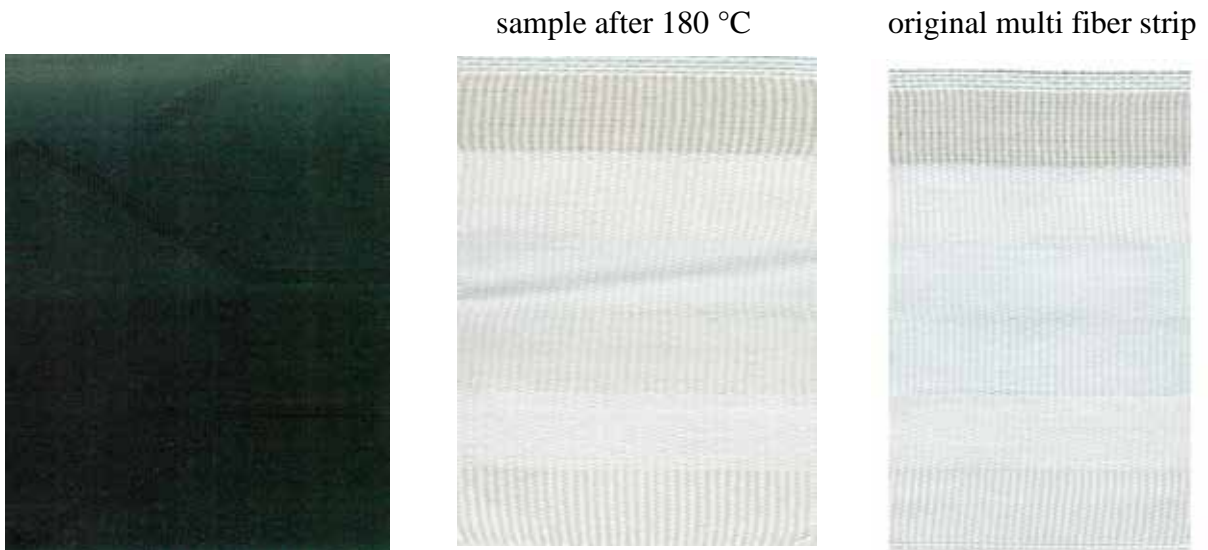


Figure 279: aNIR® Dianix® Black XF2 300% dark shade (setup 1) sample after 180 °C

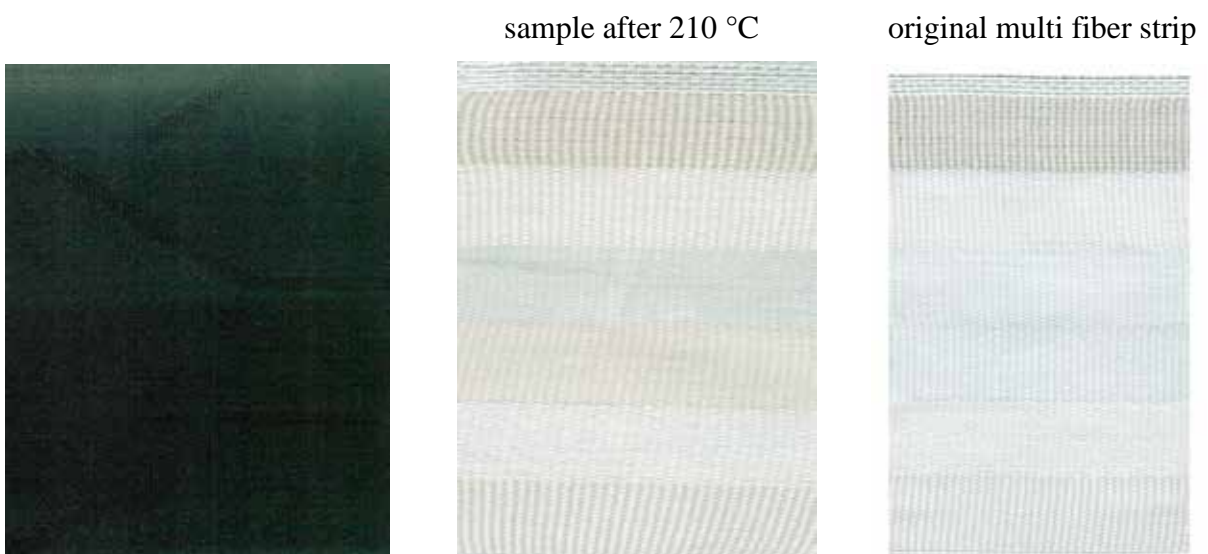


Figure 280: aNIR® Dianix® Black XF2 300% dark shade (setup 1) sample after 210 °C

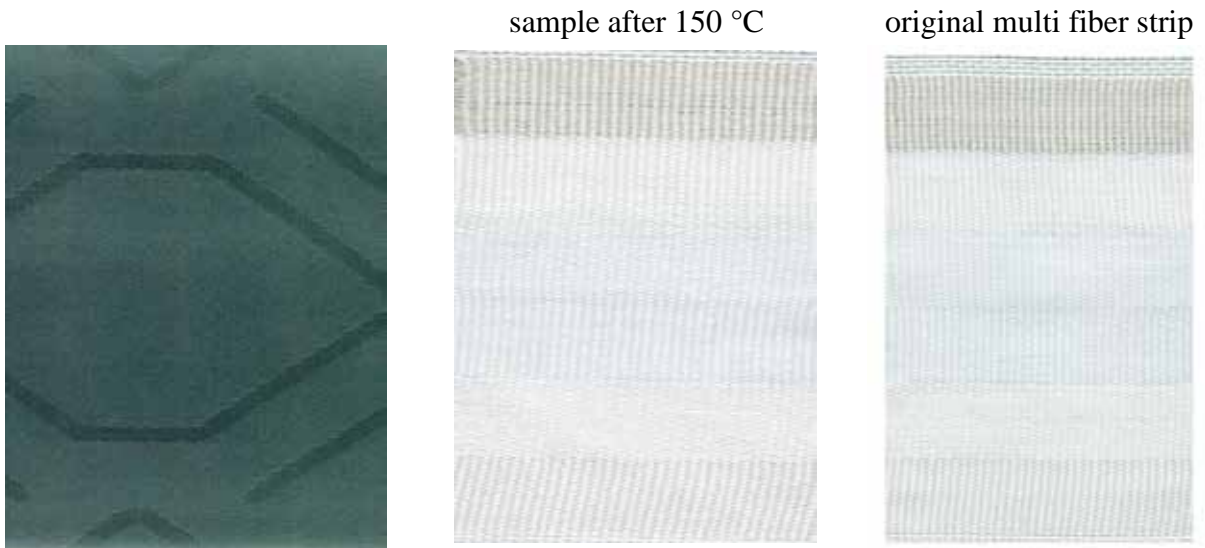


Figure 281: aNIR® Dianix® Black XF2 300% pale shade (setup 2) sample after 150 °C

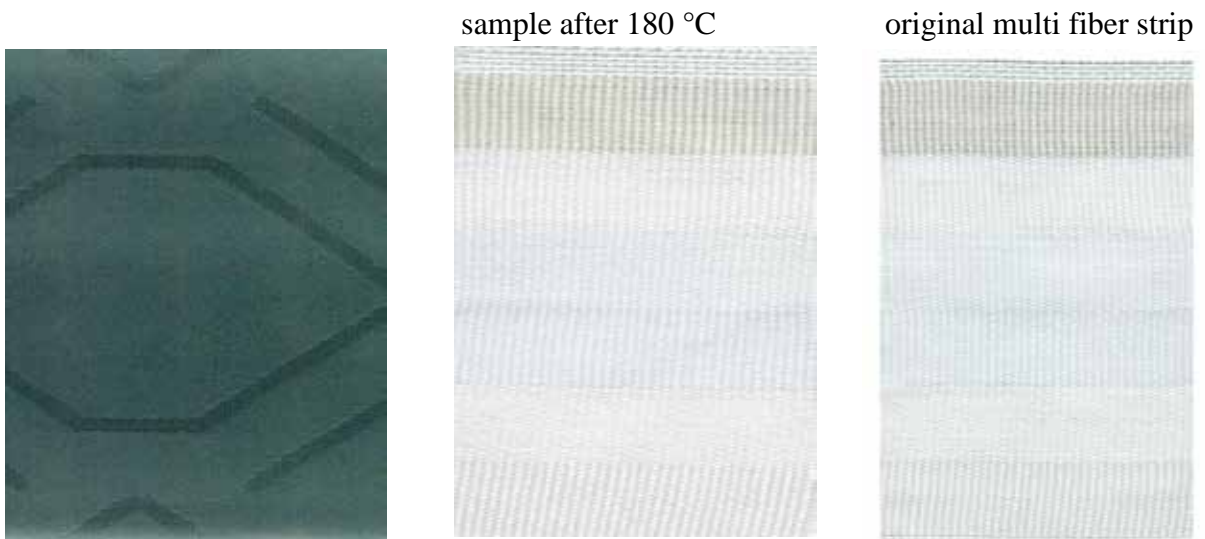


Figure 282: aNIR® Dianix® Black XF2 300% pale shade (setup 2) sample after 180 °C



Figure 283: aNIR® Dianix® Black XF2 300% pale shade (setup 2) sample after 210 °C



Figure 284: aNIR® Dianix® Black XF2 300% medium shade (setup 2) sample after 150 °C



Figure 285: aNIR® Dianix® Black XF2 300% medium shade (setup 2) sample after 180 °C

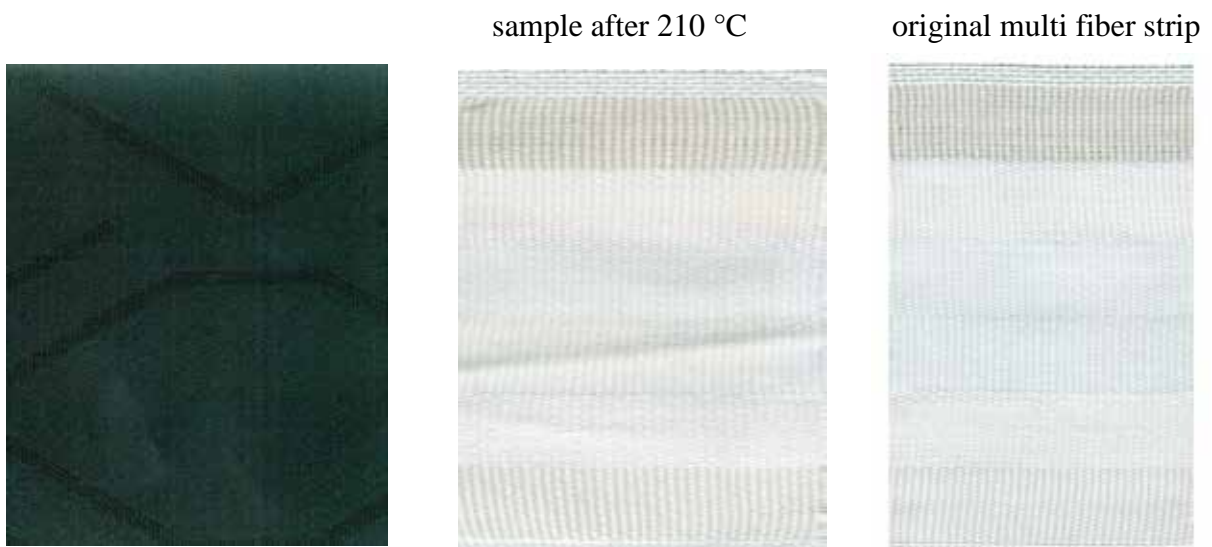


Figure 286: aNIR® Dianix® Black XF2 300% medium shade (setup 2) sample after 210 °C

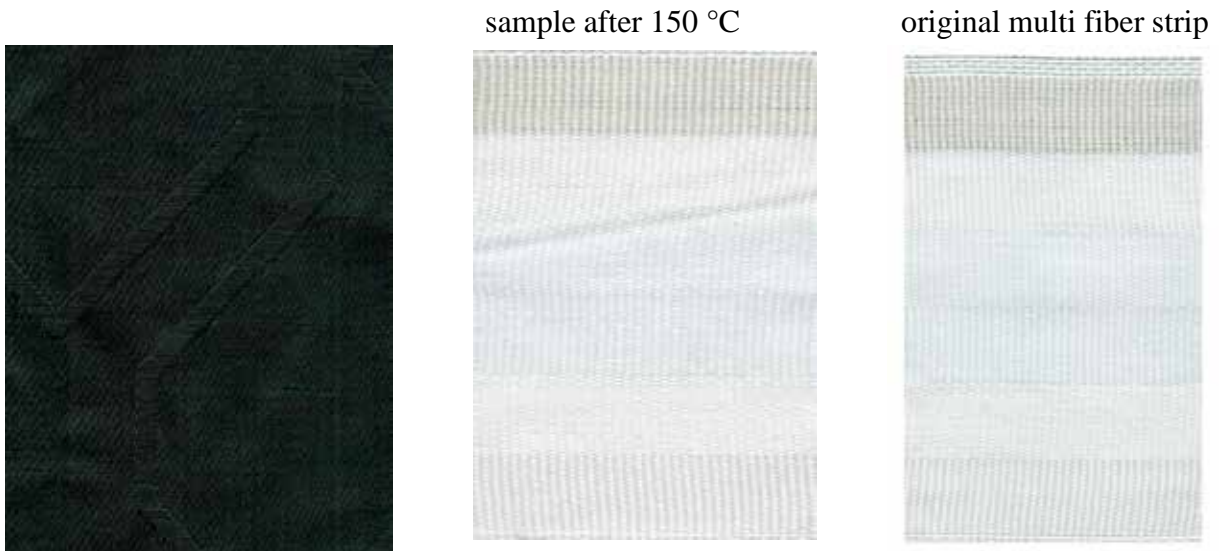


Figure 287: aNIR® Dianix® Black XF2 300% dark shade (setup 2) sample after 150 °C

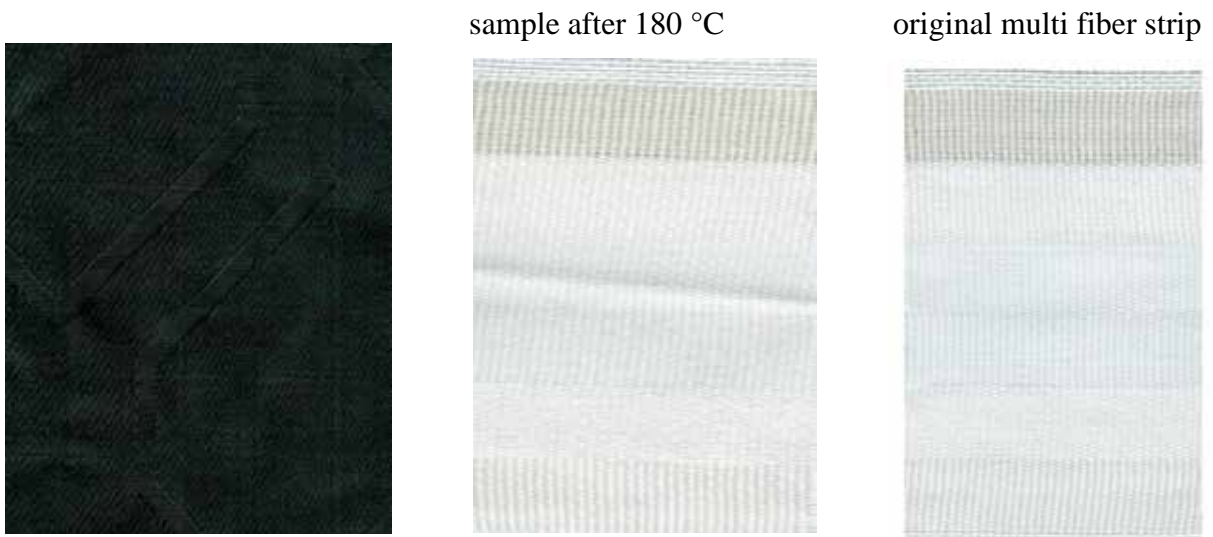


Figure 288: aNIR® Dianix® Black XF2 300% dark shade (setup 2) sample after 180 °C



Figure 289: aNIR® Dianix® Black XF2 300% dark shade (setup 2) sample after 210 °C



Figure 290: aNIR[®] Dianix[®] Black CC-R pale shade (setup 2) sample after 150 °C

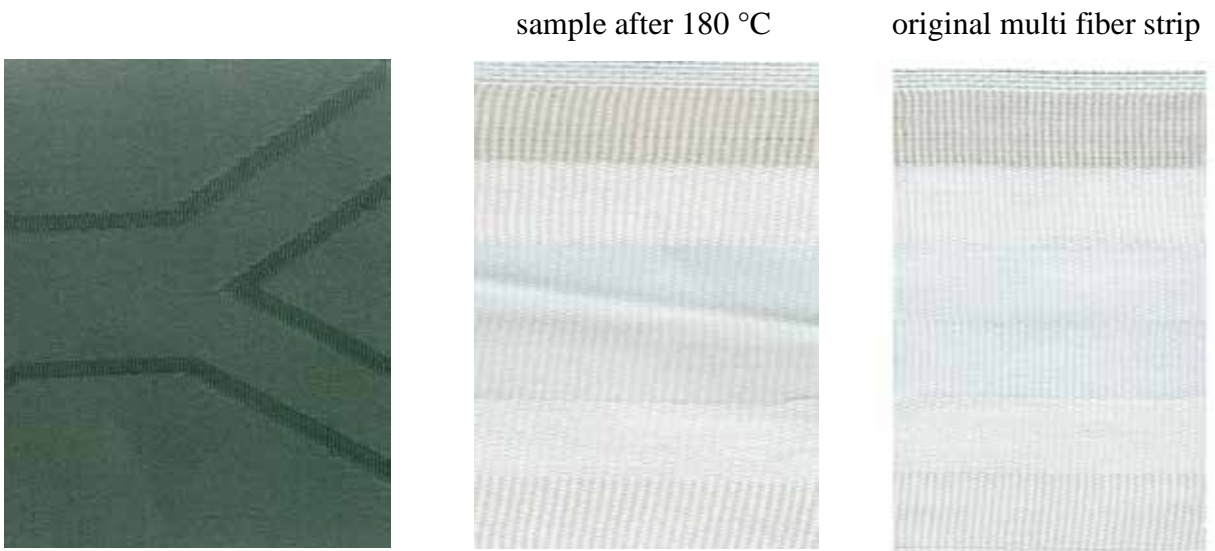


Figure 291: aNIR[®] Dianix[®] Black CC-R pale shade (setup 2) sample after 180 °C

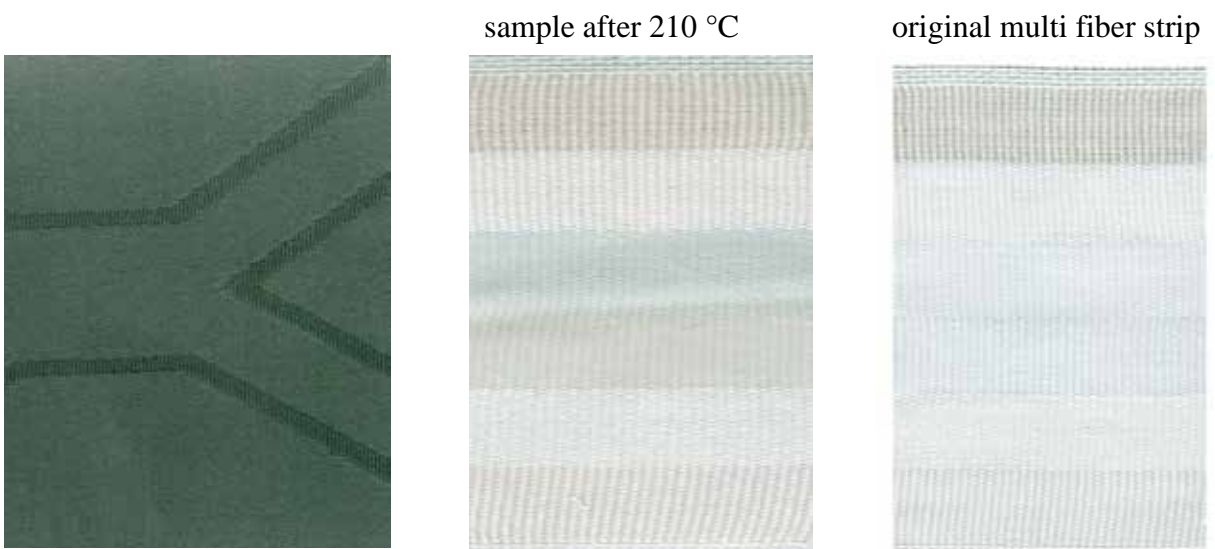


Figure 292: aNIR[®] Dianix[®] Black CC-R pale shade (setup 2) sample after 210 °C



Figure 293: aNIR® Dianix® Black CC-R medium shade (setup 2) sample after 150 °C

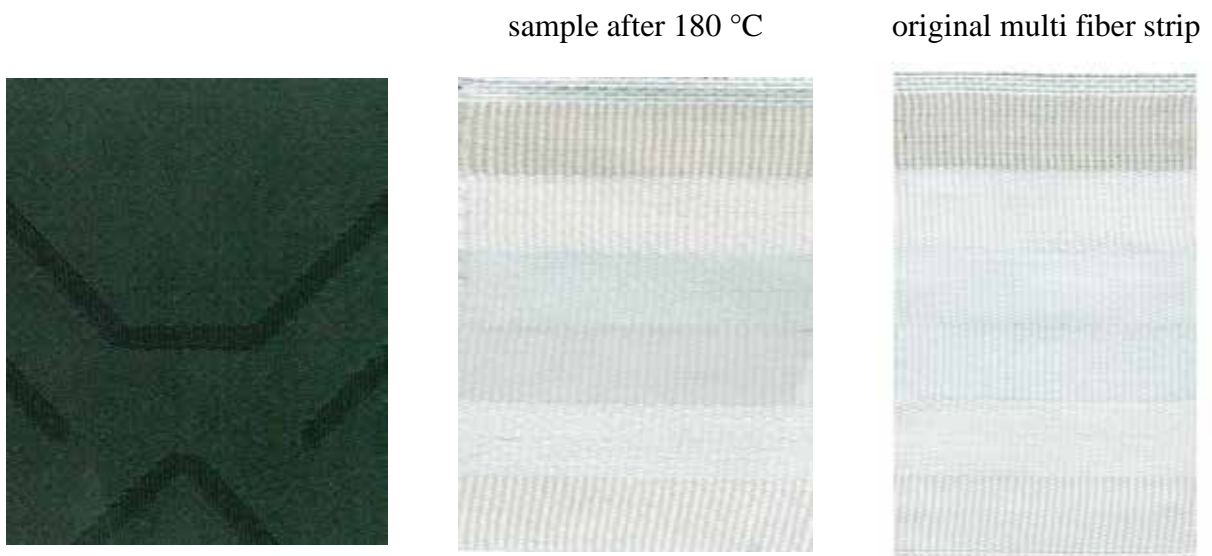


Figure 294: aNIR® Dianix® Black CC-R medium shade (setup 2) sample after 180 °C

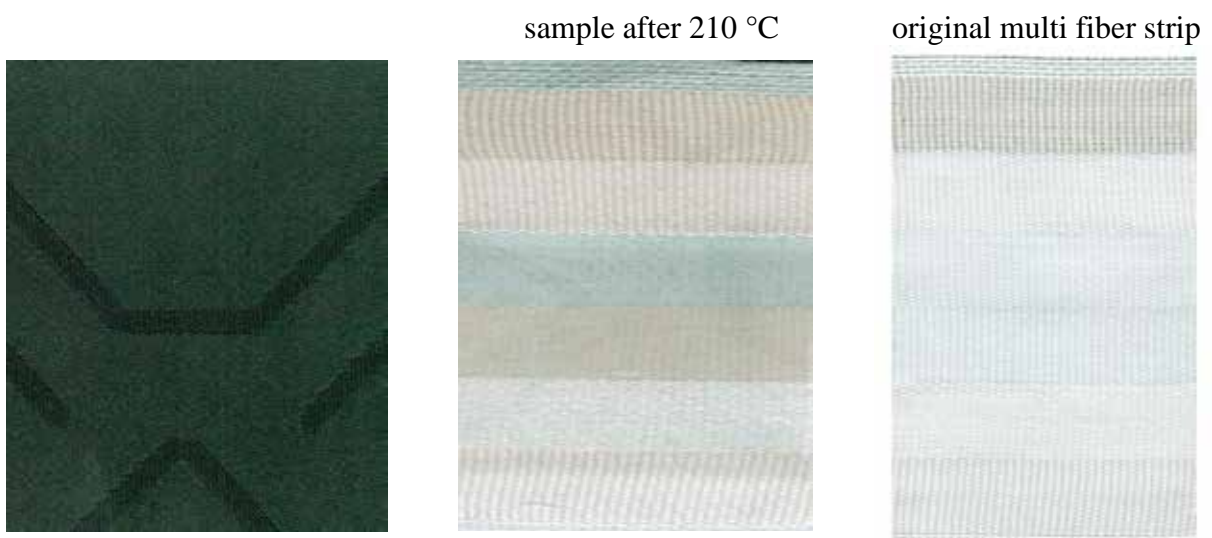


Figure 295: aNIR® Dianix® Black CC-R medium shade (setup 2) sample after 210 °C

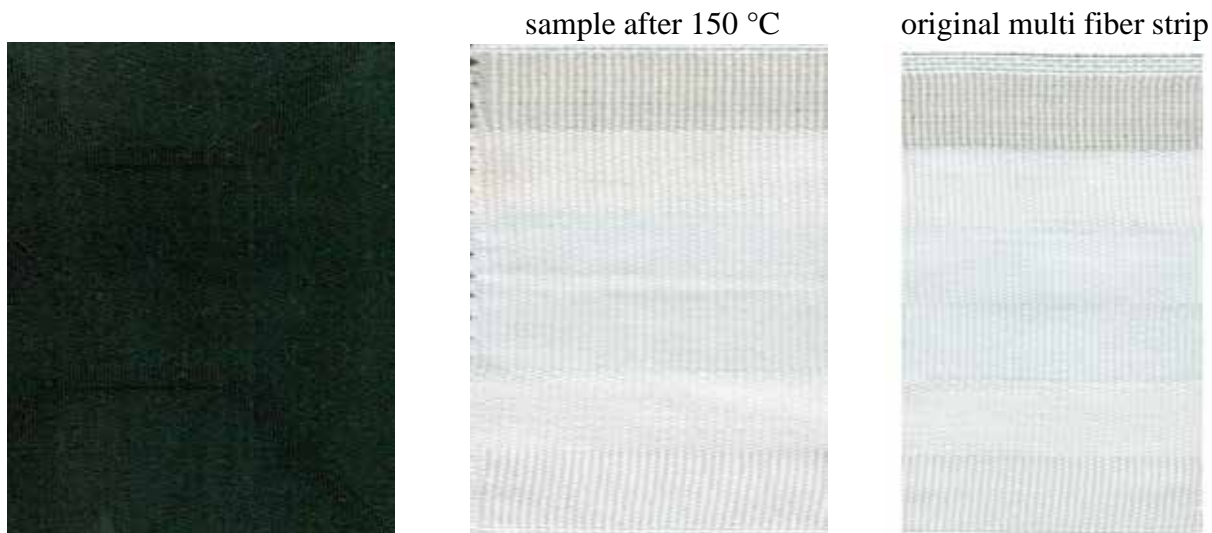


Figure 296: aNIR® Dianix® Black CC-R dark shade (setup 2) sample after 150 °C



Figure 297: aNIR® Dianix® Black CC-R dark shade (setup 2) sample after 180 °C



Figure 298: aNIR® Dianix® Black CC-R dark shade (setup 2) sample after 210 °C

Affidavit

I confirm that I have produced this thesis by myself and without using resources other than those mentioned. All ideas taken directly or indirectly from other publications are indicated. At my best knowledge, there was no thesis publicized or submitted to any examination authority before which is equal or similar to this thesis.

Erklaerung

Ich erkläre hiermit, dass ich die vorliegende Arbeit selbstständig und ohne Benutzung anderer als der angegebenen Hilfsmittel angefertigt habe; die aus fremden Quellen direkt oder indirekt uebernommenen Gedanken sind als solche kenntlich gemacht. Die Arbeit wurde nach meiner besten Kenntnis bisher in gleicher oder aehnlicher Form keiner anderen Pruefungsbehoerde vorgelegt und auch noch nicht veroeffentlicht.

Hof, 25.11.2022

Antwi Andy Buobu,

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