

The impact of water released from boehmite nanoparticles during curing in epoxy-based nanocomposites

Supporting Information

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Powder X-ray diffraction (XRD)

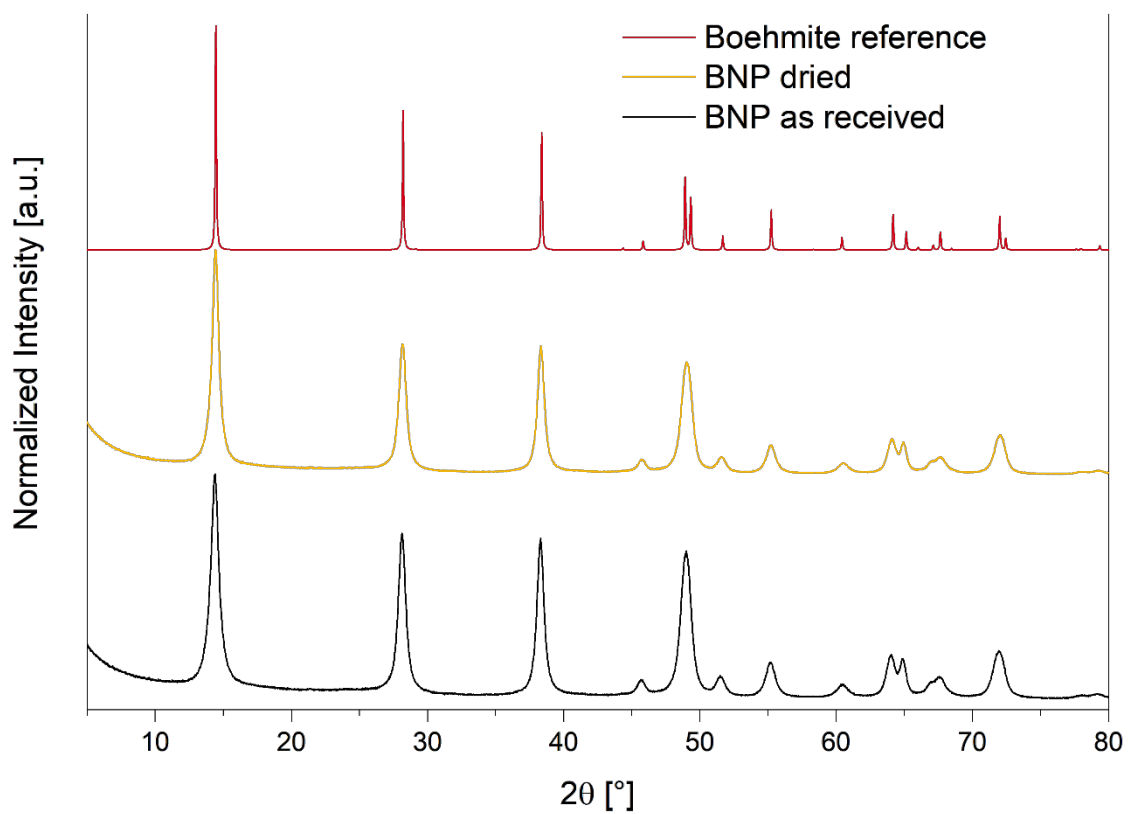


Figure S1 X-ray diffractograms of dried and as received BNP.

XRD data shows that the crystalline structure of the dBNP is not affected by drying at 80 °C for 5 h.

FTIR spectra

The following spectra give an overview on the changes by heating at 80 °C of AH or Acc - pure, with H₂O, with boehmite nanoparticles (BNP) or with dried BNP (dBNP). The measurements after 15 and 90 min have been recorded are not shown to keep the diagrams well-structured. They fit with the observable trends and do not contain further information.

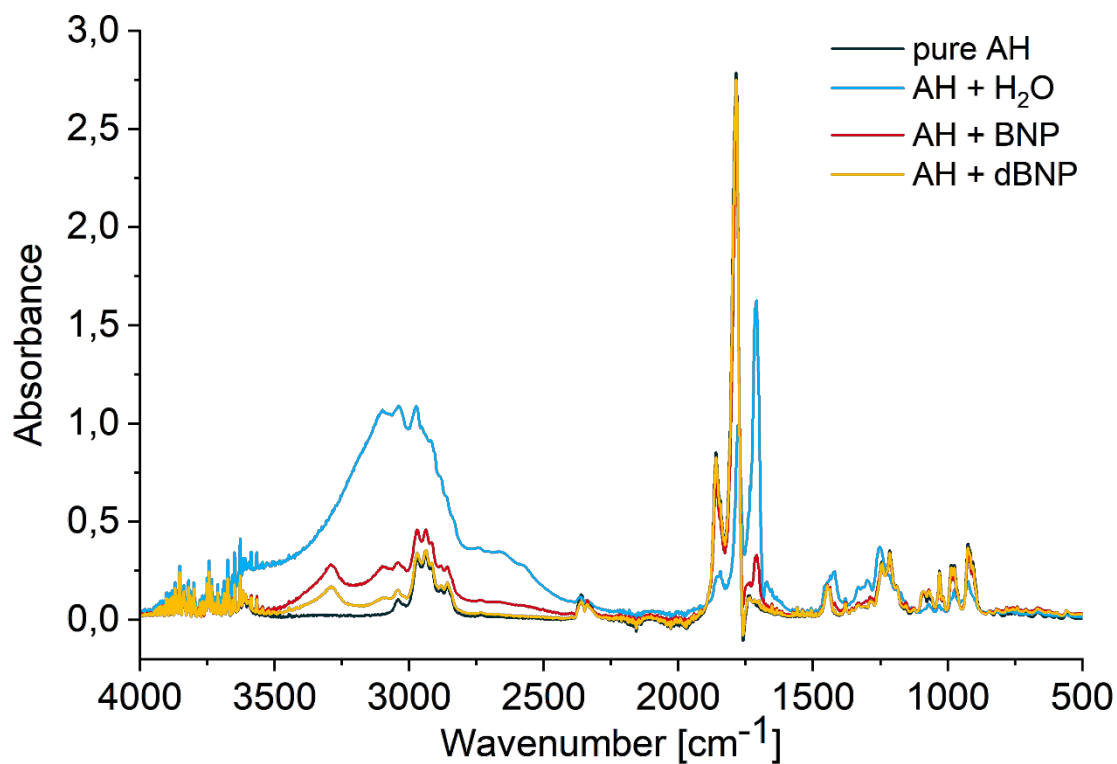


Figure S2 Full ATR-FTIR spectra of heated (80 °C, 30 min) anhydride hardener pure (black), with water (blue), BNP (red) or dBNP (yellow). Suggesting a reaction between water released from the BNP and the AH. Due to phase separation only the signals of AH and hydrolyzed AH are visible for AH + H₂O and no signals of water.

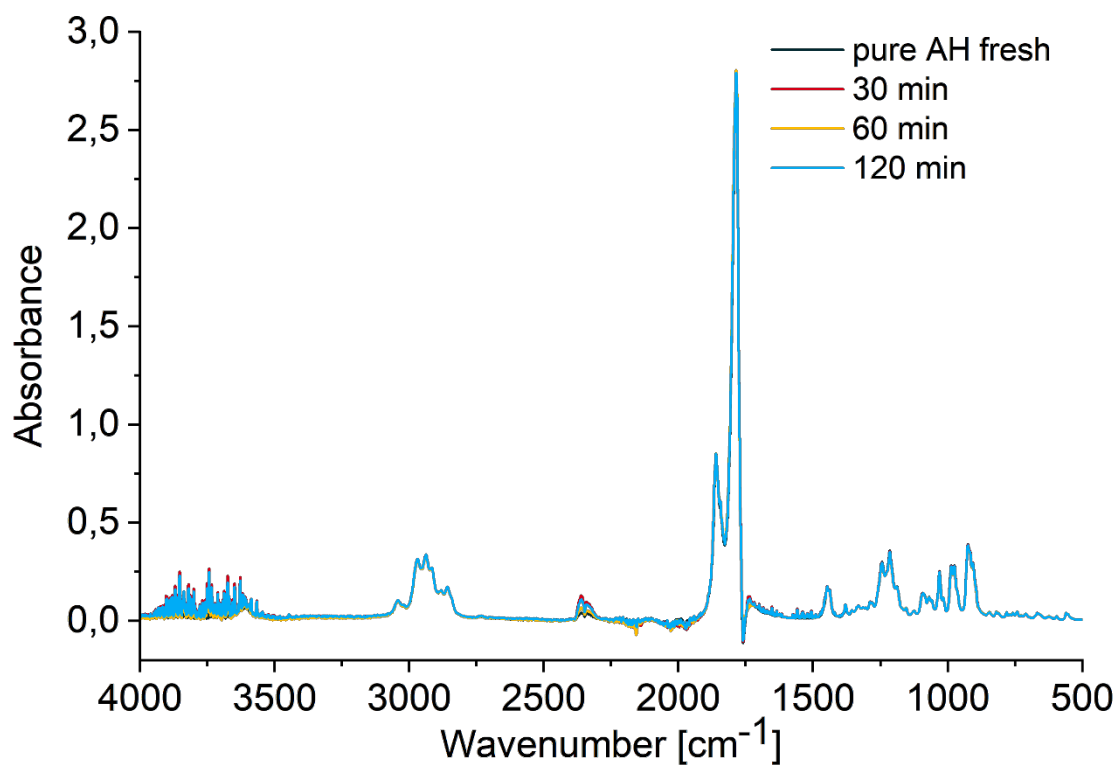


Figure S3 ATR-FTIR spectra of heating series (80 °C) for pure anhydride hardener (AH). No changes visible.

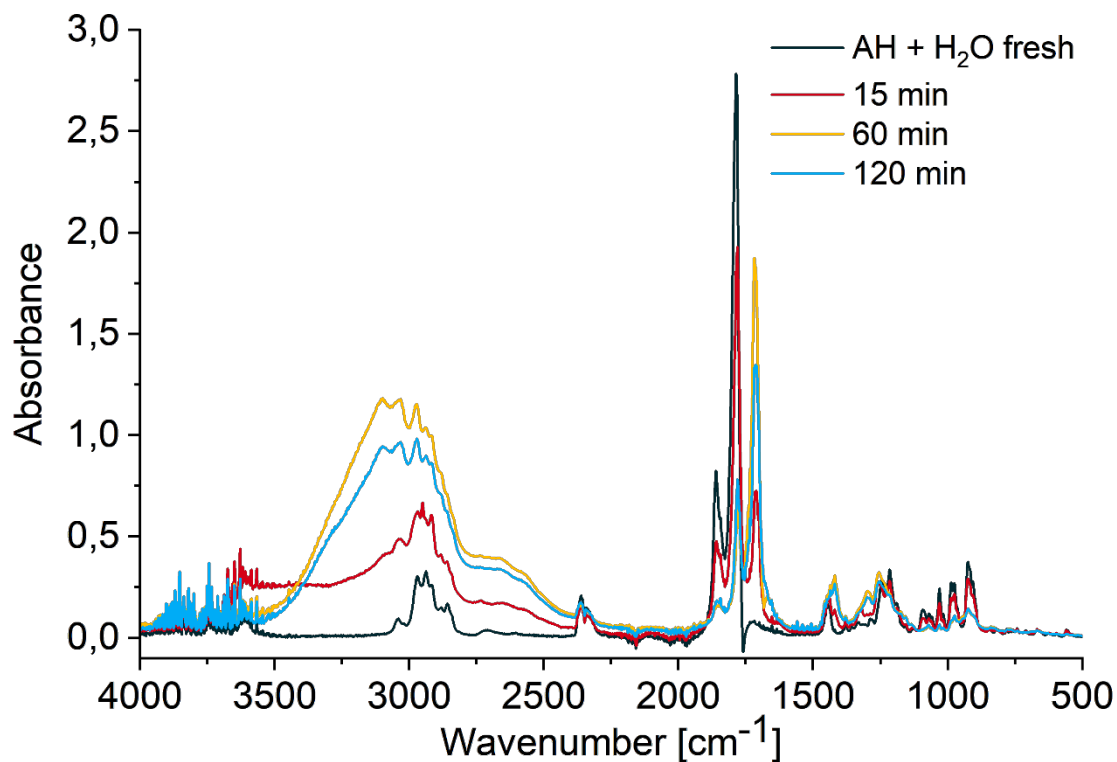


Figure S4 ATR-FTIR spectra of heating series (80 °C) for AH + H₂O. Hydrolysis of the AH is conclusive (see Figure 3). Additional signals appear in the carbonyl-region (1715 cm⁻¹) and for the hydroxyl-groups around 1440 cm⁻¹ and 3200 cm⁻¹. Due to phase separation only the signals of AH and hydrolyzed AH are visible and no signals of water.

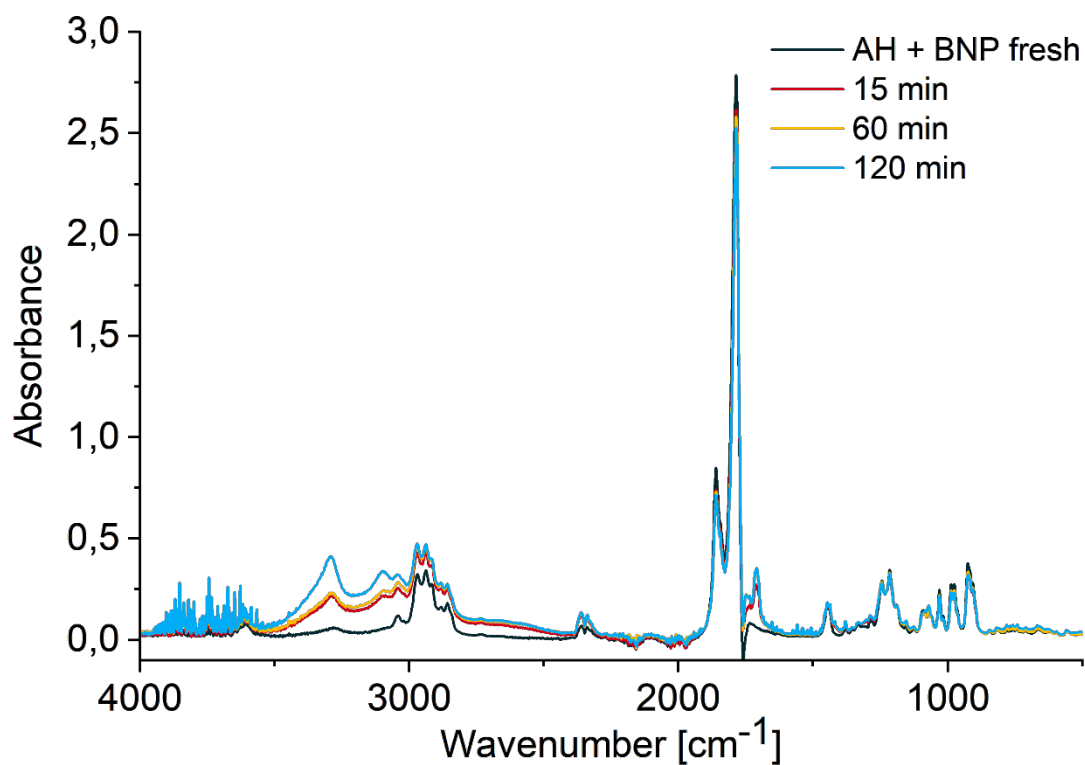


Figure S5 ATR-FTIR spectra of heating series (80 °C) for AH + BNP. Hydrolysis of the AH by water released from the BNP is conclusive (see Figure 3). Additional signals are visible in the carbonyl-region (1715 cm⁻¹).

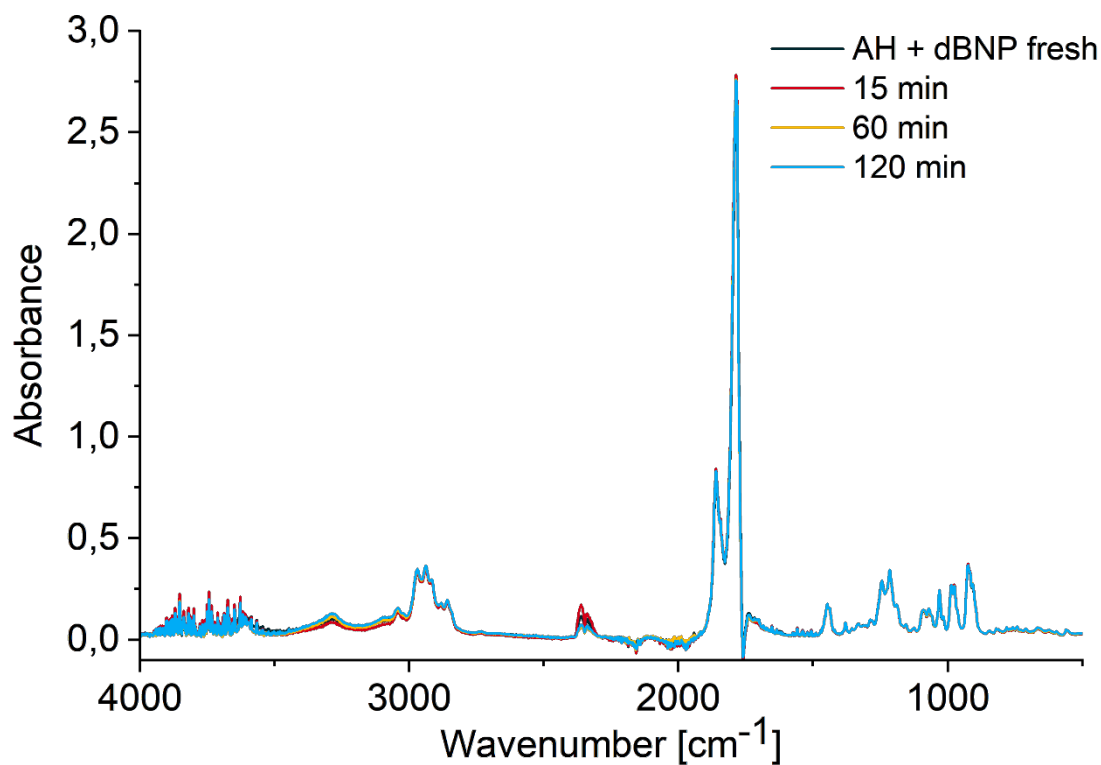


Figure S6 ATR-FTIR spectra of heating series (80 °C) for AH + dBNP. No changes visible.

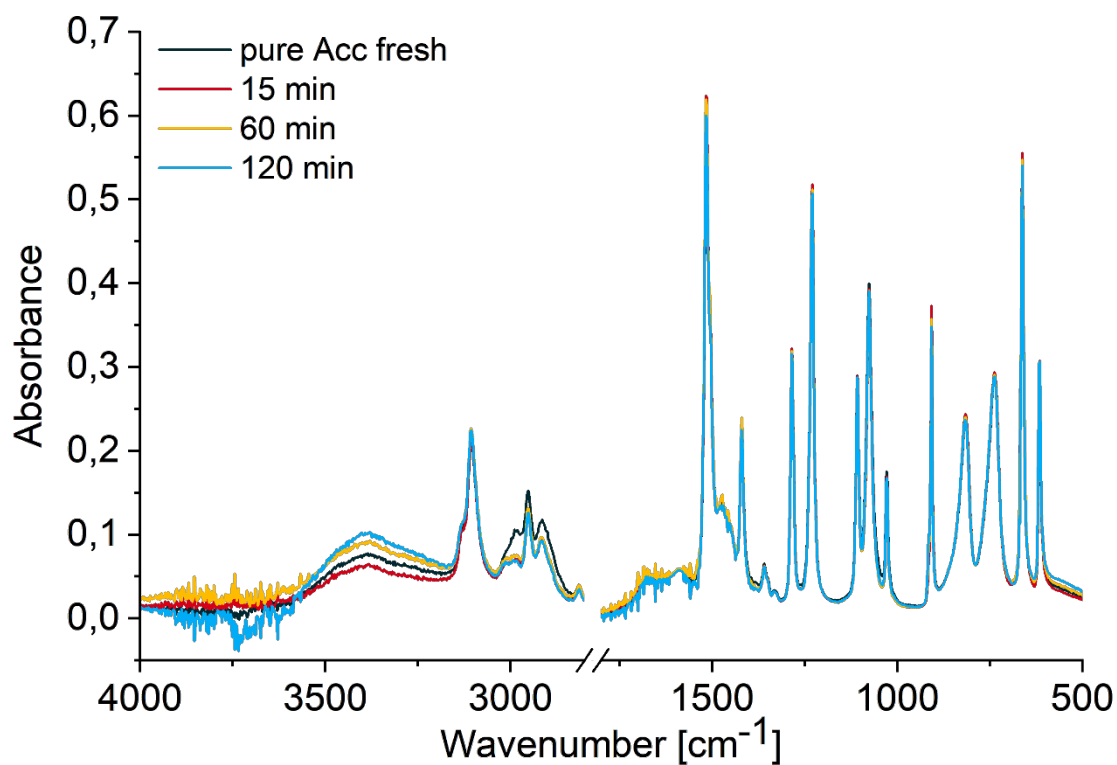


Figure S7 ATR-FTIR spectra of heating series (80 °C) for pure amine accelerator (Acc). Minor changes appear due to reaction with humidity. (changed signals see table 2)

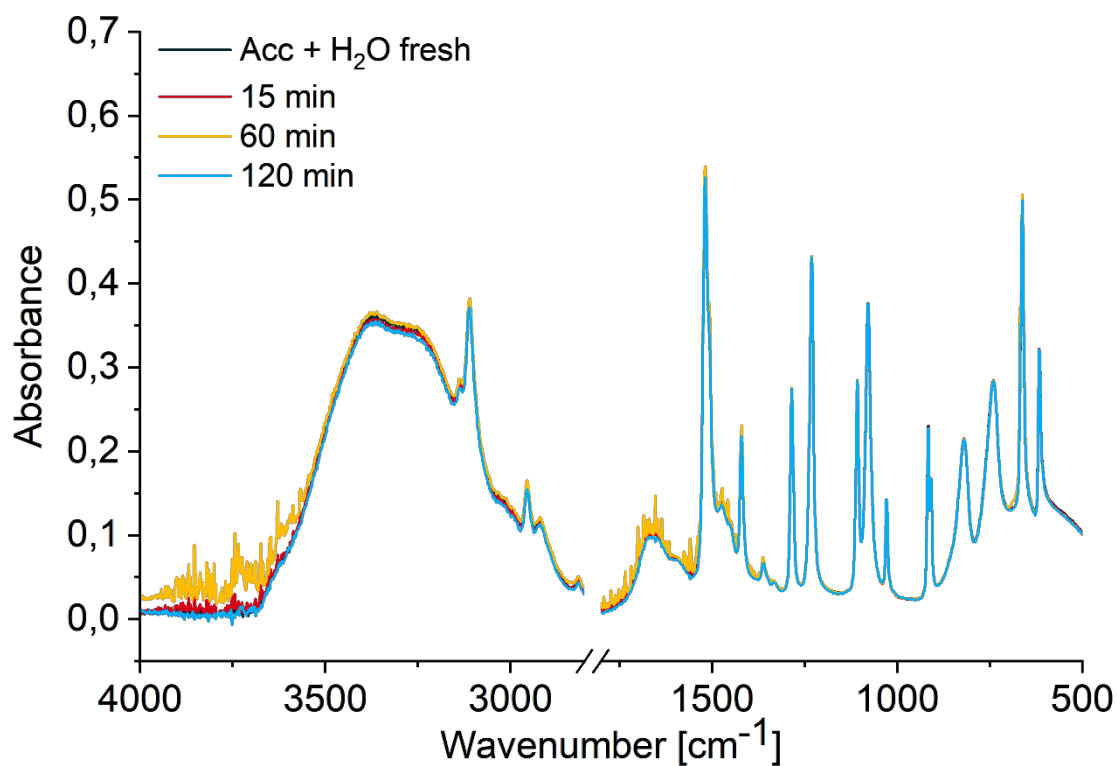


Figure S8 ATR-FTIR spectra of heating series (80 °C) for Acc + H₂O. The partly protonation of Acc by water is visible. (changed signals see table 2)

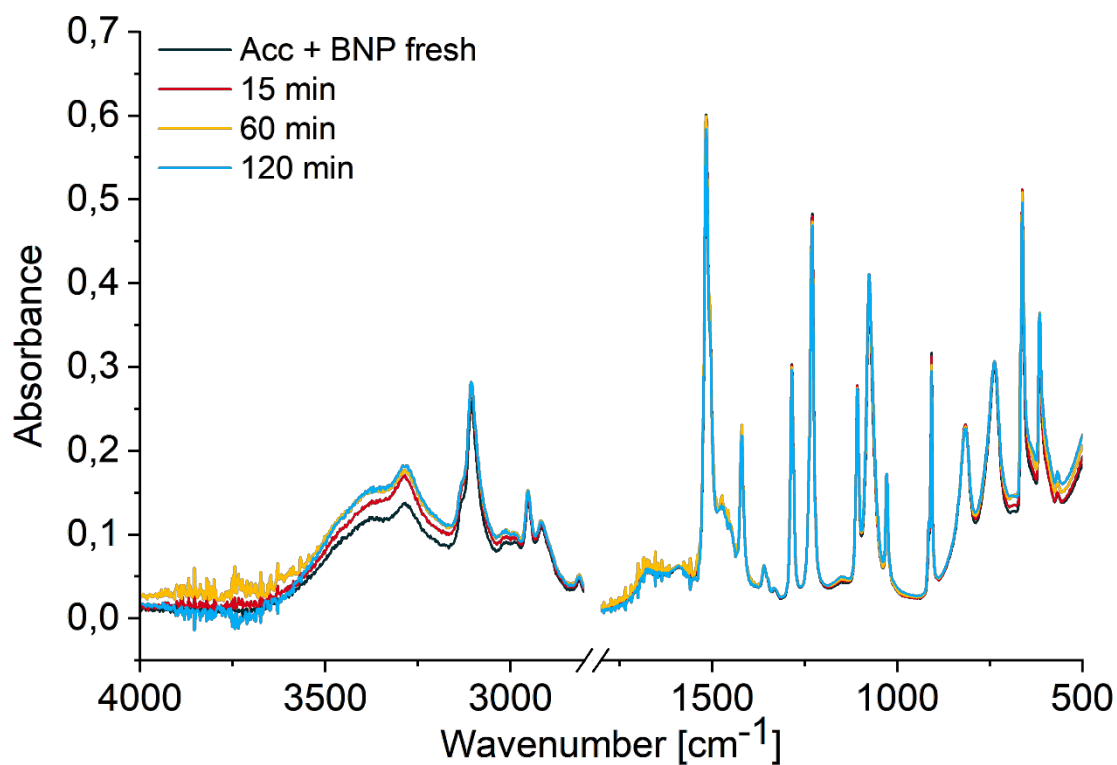


Figure S9 ATR-FTIR spectra of heating series (80 °C) for Acc + BNP. The partly protonation of Acc by water released from the BNP is visible. (changed signals see table 2)

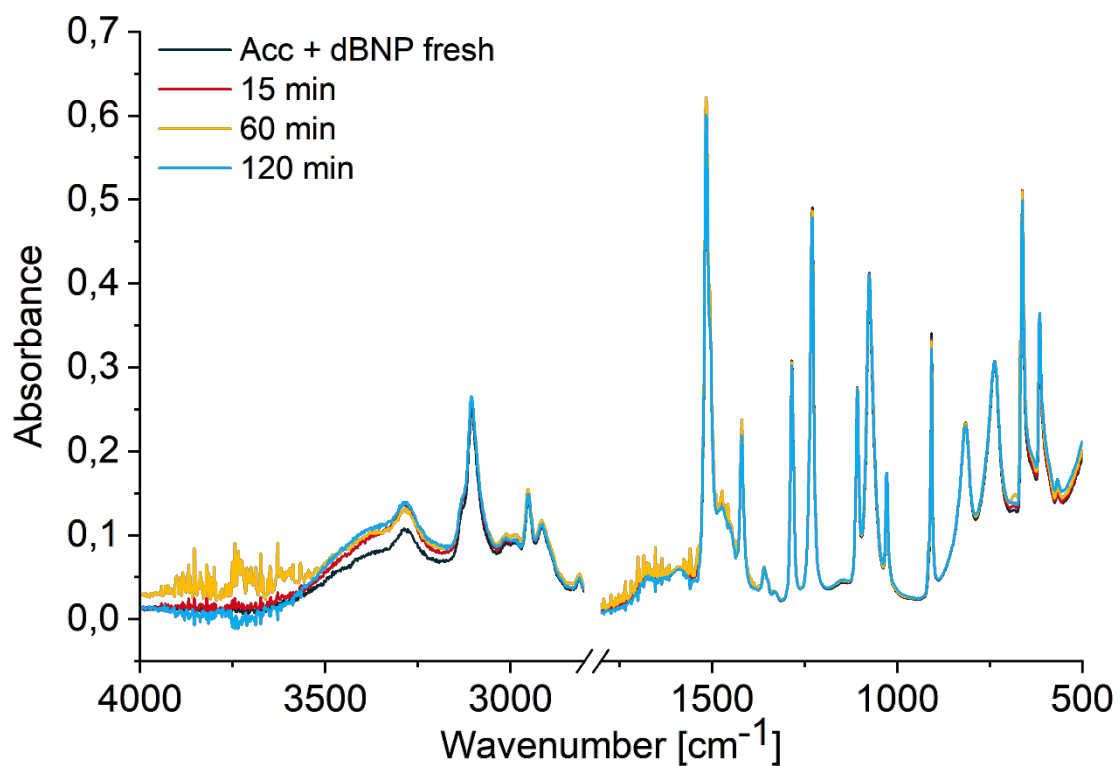


Figure S10 ATR-FTIR spectra of heating series (80 °C) for pure Acc + dBNP. Minor changes appear due to reaction with humidity. (changed signals see table 2)

NIR spectroscopy on DGEBA

NIR-spectra have been recorded between 7500 cm^{-1} and 4000 cm^{-1} . The epoxy groups which are most likely to react show characteristic absorption bands from 4552 to 4497 cm^{-1} and 6149 to 6026 cm^{-1} . They are commonly used to observe the conversion of the DGEBA during curing reaction.^[1] Any influence of the BNP or water as mentioned for mid-infrared lead at least lead to a reduction of the peak intensities e.g. due to oligomerization or hydrolysis of the DGEBA.

[1] E. Duemichen, M. Javdanitehran, M. Erdmann, V. Trappe, H. Sturm, U. Braun, G. Ziegmann, *Thermochimica Acta* **2015**, *616*, 49–60.

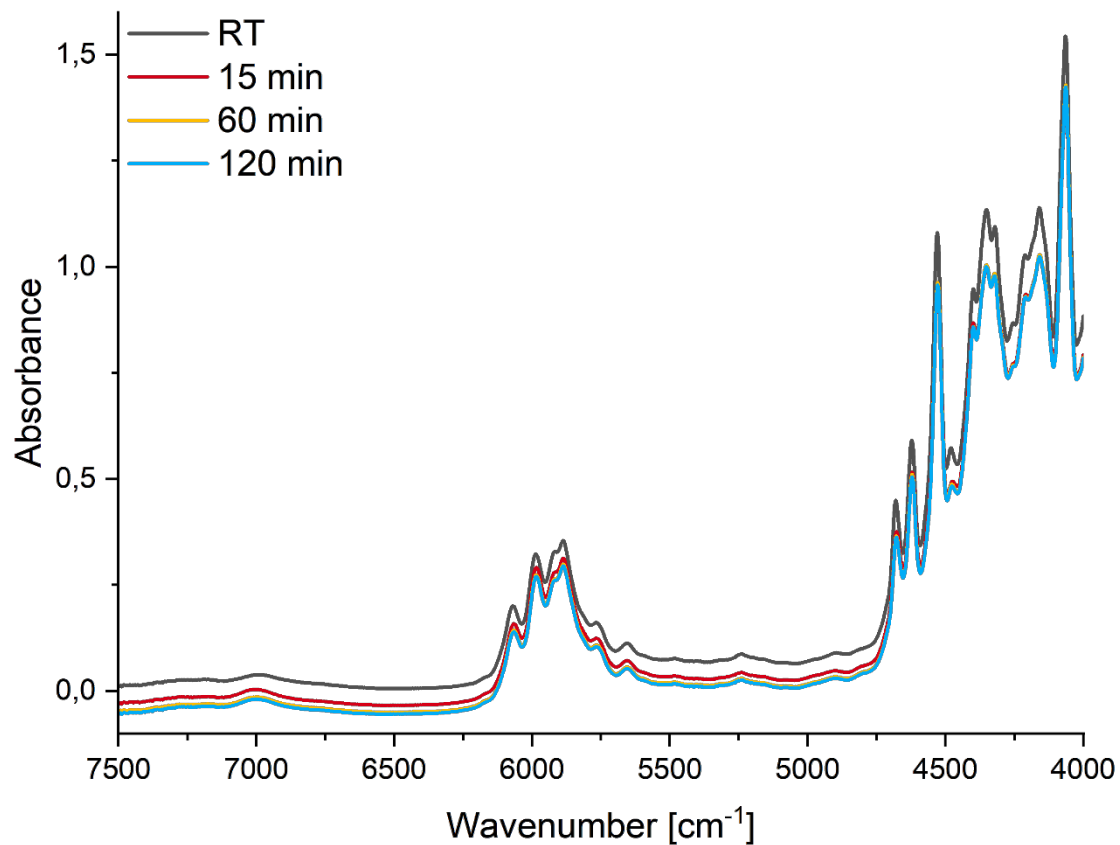


Figure S11 NIR spectra of heating (80 °C) for pure DGEBA. No change of the signals observable. Only a baseline shift by heating from room temperature (RT, 22 °C) to 80 °C.

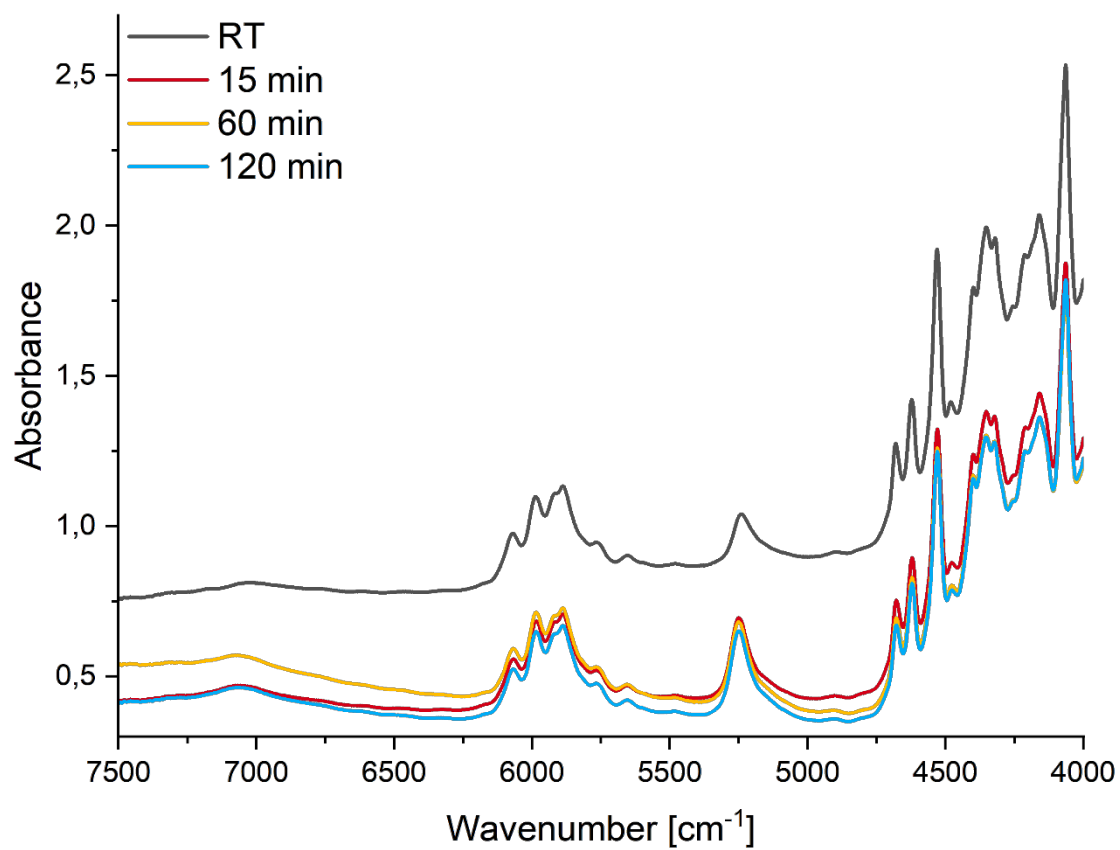


Figure S12 NIR spectra of heating (80 °C) for DGEEBA with 30 wt.% water. No change of the signals observable. Only a baseline shift by heating from room temperature (RT, 22 °C) to 80 °C.

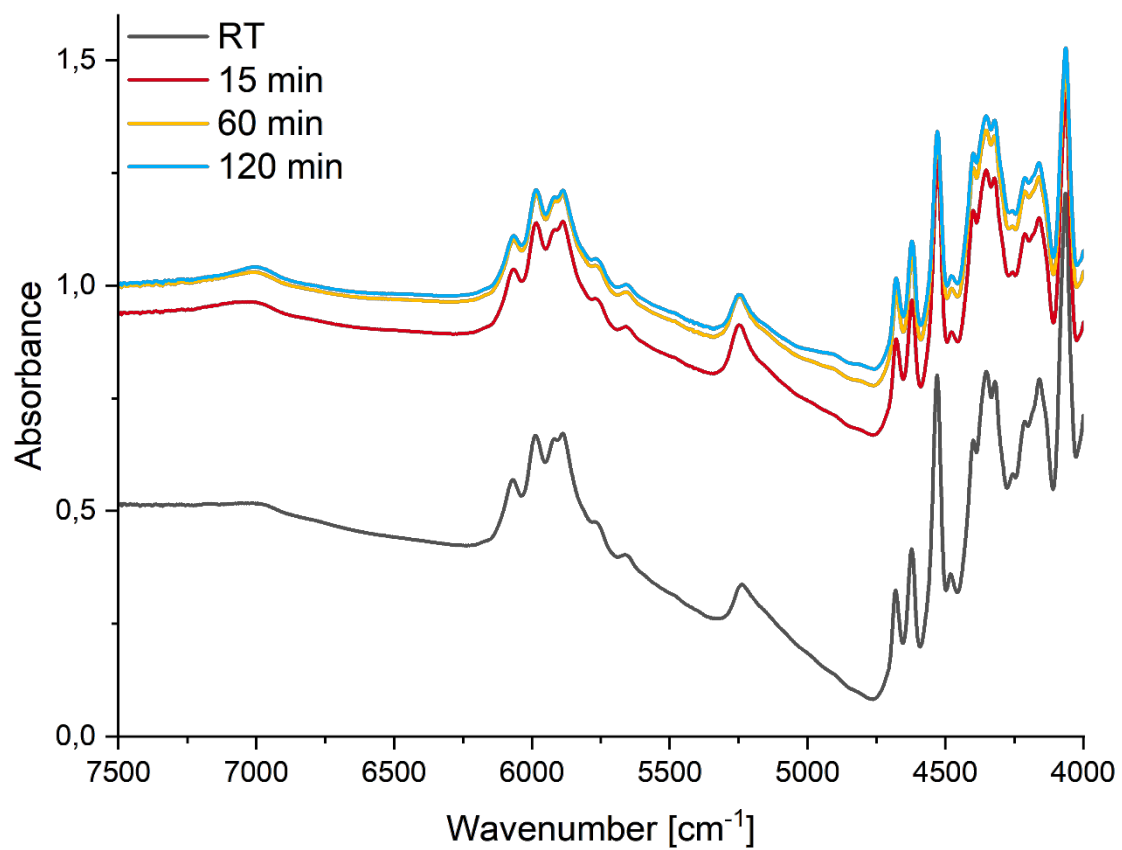


Figure S13 NIR spectra of heating (80 °C) for DGEBA with 30 wt.% BNP. No changes of the peaks observable. Only a baseline shift by heating from room temperature (RT, 22 °C) to 80 °C.

The following diagram shows the full DRIFT spectra of Ep and Ep + 15BNP. The significant change in the carbonyl region is depicted in Figure 6 of the main document.

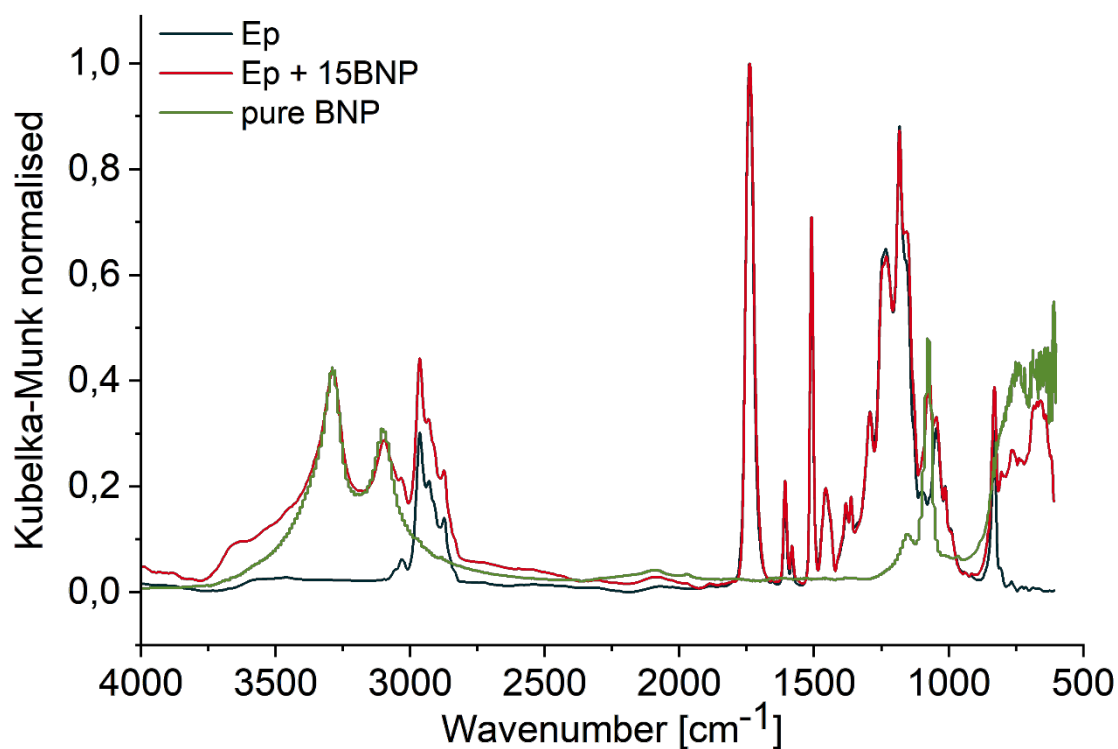


Figure S14 DRIFT spectra of cryomilled samples in KBr: cured epoxy polymer pure (Ep, black), nanocomposite with 15 wt.% BNP (Ep + 15 BNP, red) and pure BNP (green). Obvious differences between Ep and Ep + 15BNP are related to the BNP. Most important difference is depicted in Figure 6.