

Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS) Analysis

The samples were also characterized by scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) analysis. The instrument used was a dual beam scanning electron/focused ion beam system (SEM/FIB LYRA I XMU, TESCAN), equipped with EDX detector (Quantax 200, Bruker). Prior to the SEM observation, the samples were coated with carbon using EMITECH K450X coating system. The measurements were performed by using an acceleration voltage of 30 kV. EDS spectra directly revealed the presence of the atomic elements in the sample. The system was left to automatically detect the elements and the following elements were determined in this way Ca, P, O and C. Manually we have checked if one from the following also presents: K, Cl and N. Only N was registered in small amount coming from the PAAm.

In Figure S1 A is shown the morphology of the sample area that was scanned. In Figure S1 B is shown the availability of the automatically detected by the system elements Ca, P and O (C being excluded as the coating is with C). The mapping of this area resulted in the elemental spectra shown in Figure S1C. The maps for all of these 3 elements distribution in the scanned area is presented in Figure S1 D. The coincidence of these 3 elements mappings spectra confirms that they are forming a joint compound.

We have used voltage of 30 kV as for polymers it means that the beam penetration is 12 000 nm. Thus the results are representative for quite a big volume from the sample as the mapping is done for surface area of 5 μm^2 (the area is enclosed in green line, Figure S1A).

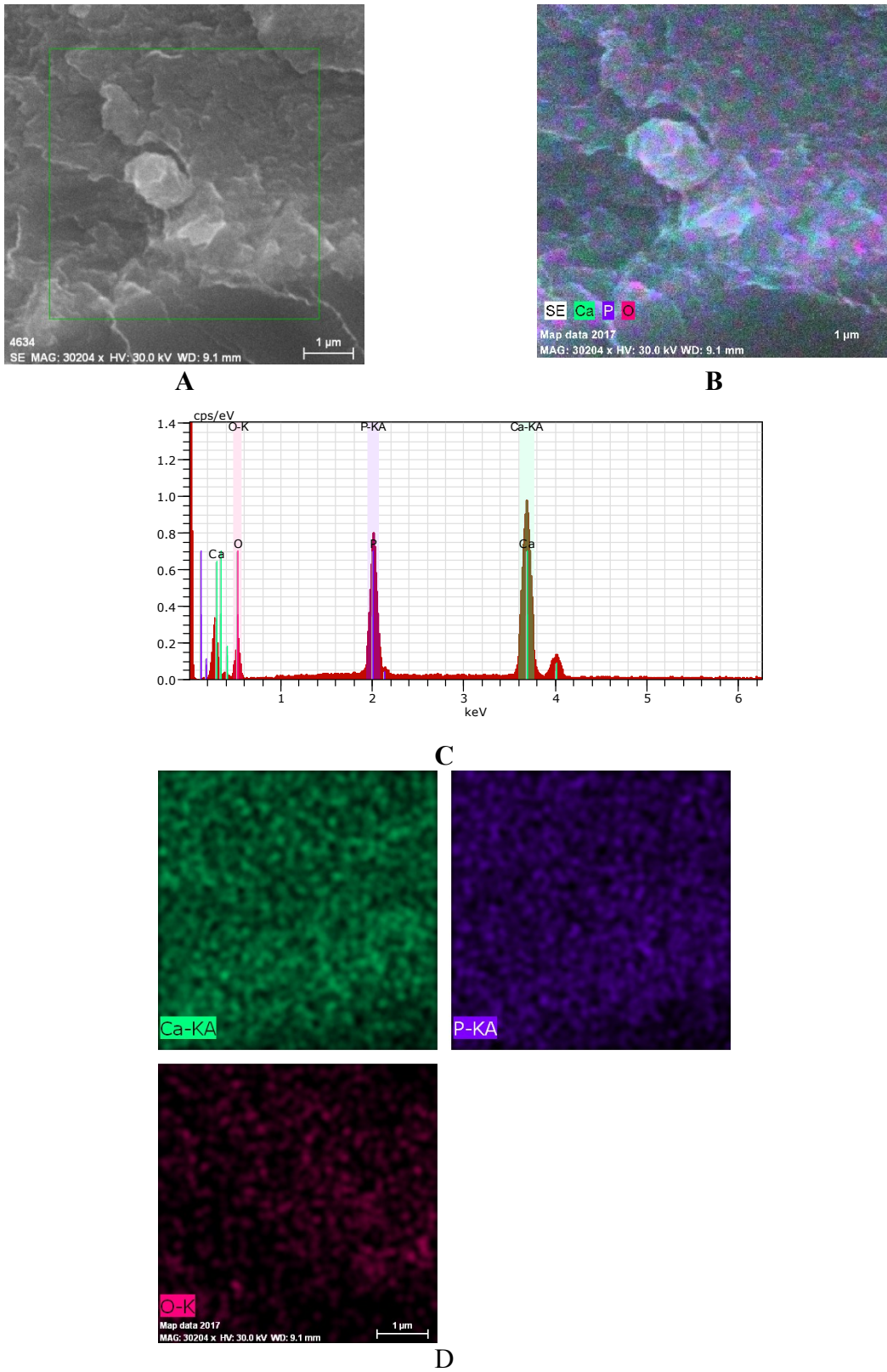


Figure S1. SEM EDX mapping for IPN 9 ($\phi^{\text{PAA}}=0.68$) with *in situ* precipitated CP.

Transmission electron microscopy

The hybride IPNs+CP materials were heated up to 900°C in order to remove (decompose) the polymer part and then the remaining inorganic (CP) part was suspended in water and dropped on a copper grid. When dried, the grids were placed and examined by TEM (JEOL-2100, 200 kV) with energy dispersive X-ray analysis (EDX). The small size (below 100 nm) of CPs precipitated in situ in IPN could be seen.

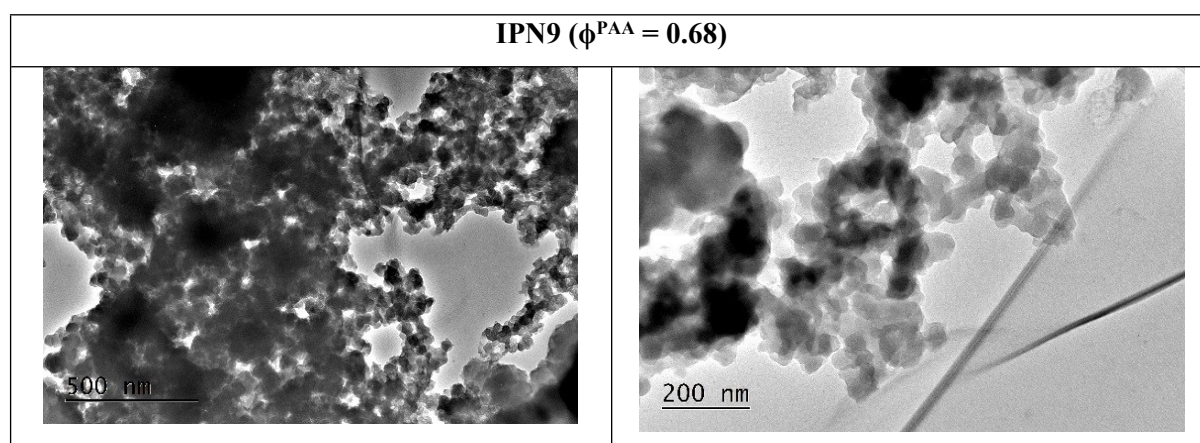


Figure S2. TEM analysis of the ashes, left after burning the sample IPN 9 ($\phi^{PAA}=0.68$) with *in situ* precipitated CP.

Infrared spectroscopy

IR spectra of PAAm and PAAm+CPs

Both spectra of *SN PAAm* and the composite material *PAAm+CPs* are quite similar as they both contain the typical for PAAm bands [24, 25]:

Table S1. Bands in the IR spectra of *PAAm+CPs* and *PAAm*, typical for PAAm.

Band, cm ⁻¹	Attribution
~ 3340 cm ⁻¹	the amide N–H asymmetric stretch in NH ₂
~3200 cm ⁻¹	the amide N–H symmetric stretch in NH ₂
~2930 cm ⁻¹	the C–H asymmetric stretch in CH ₂
~1655 cm ⁻¹	the amide C=O stretch
~1610 cm ⁻¹	the NH bend
doublet at 1413 cm ⁻¹ and 1453 cm ⁻¹	the deformation vibration of –NH groups
~1320 cm ⁻¹	the C–H deformation
doublet at 1320 and 1349 cm ⁻¹	the stretching vibration of –NH groups
~1110 cm ⁻¹	C–O stretching vibration

In both IR spectra (*PAAm* as well as *PAAm+CP*) a broad band in the region 3000-3700 cm⁻¹ appears due to the OH stretching vibrations of absorbed water, hydroxyl group being H-bound as the peak is very broad.

IR spectra of PAA and PAA+CPs

The IR spectra of *SN PAA* and *PAA* with *in situ* deposited CP (designated as *PAA+CP*) again have quite many common bands [23, 24]:

Table S2. Bands in the IR spectra of *PAA+CPs* and *PAA*, typical for *PAA*.

Band, cm⁻¹	Attribution
~2924 cm ⁻¹	the typical C–H asymmetric stretch in CH ₂
~1700 cm ⁻¹	the typical C=O stretching vibration of carbonyl group in <i>PAA</i> , here shifted to lower frequencies in the neat <i>PAA</i> (it is usually above 1712 cm ⁻¹) which could be due to H-bonding
~1450 cm ⁻¹	$\delta_{\text{CH}_2} + \delta_{\text{O-H in the plane}} + \nu_{\text{C-O}}$
~1402 cm ⁻¹	$\delta_{\text{CH}_2} + \delta_{\text{O-H in the plane}} + \nu_{\text{C-O}}$
~1030 cm ⁻¹ and 1114 cm ⁻¹	the C–O stretch typical for carboxylic esters or salts
~798 cm ⁻¹ .	$\delta_{\text{O-H in the plane}}$

In both IR spectra (*PAA* as well as *PAA+CP*) a broad band in the region 3000-3700 cm⁻¹ appears due to the OH stretching vibrations of absorbed water, hydroxyl group being H-bound as the peak is very broad.