

*Ilma Cirne<sup>1</sup>, Maria Esperidiao<sup>1</sup>, Jaime Boaventura<sup>1</sup> and Elizabete Lucas<sup>2</sup>*

## PREPARATION AND CHARACTERIZATION OF COMPOUNDS BASED ON POLY(HYDROXYMETHYLACRYLAMIDE) AND POST-CONSUMER POLYPROPYLENE

<sup>1</sup>Federal University of Bahia, Institute of Chemistry,  
R. Barao Jeremoabo, 147, Ondina, 40170-115, Salvador, Brazil; [ilmaufba@gmail.com](mailto:ilmaufba@gmail.com)

<sup>2</sup>Federal University of Rio de Janeiro, COPPE/PEMM,

Av. Horacio Macedo 2030 - Ilha do Fundao, 21941598, Rio de Janeiro, Brazil; [elucas@metalmat.ufrj.br](mailto:elucas@metalmat.ufrj.br)

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**Abstract.** In this work, in order to obtain materials with potential for treatment of water from oil industry, polymer composites were synthesized by polymerization reaction *via* free radical using *n*-hydroxymethyl acrylamide (HMAA) in the presence of post-consumer polypropylene (PP) with subsequent condensation reaction catalyzed by heating, which avoids the use of crosslinking agents. The products were characterized by Fourier transform infrared spectroscopy (FT-IR), optical microscopy (OM), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and X-ray diffraction (XRD). Moreover, the bulk density and the degree of swelling were also determined. The synthesis was shown to be reproducible and led to achieving polymer composites with high levels of PP after usage from food packaging, which can be associated with a relatively low cost of production. The swelling capacity and the thermal stability of the composite increased with increasing PP content in the mixture.

**Keywords:** polymer composite, polymer blend, post-consumer polypropylene, poly(hydroxymethyl acrylamide), crosslinking reaction, polymer characterization.

### 1. Introduction

Polymer composites and nanocomposites have been prepared using different kind of materials for several purposes. Such composites can be prepared by mixing two different polymers or polymer(s) and (nano)filler [1-4]. The mixture of two homopolymers, in the majority of cases, produces an immiscible system since exhibits certain degree of phase separation [5-6].

Despite the large amount of studies involving polymer blends, a network of poly(hydroxymethyl acrylamide) (PHMAA) and polypropylene (PP) has not been described in the literature. This network can be made from *n*-hydroxymethyl acrylamide (HMAA) reaction in the presence of polypropylene, without using crosslinking agents.

Polypropylene (PP) is a thermoplastic resin easily processed with stereo regular structure that favors the development of crystalline regions which, depending on the processing conditions, allow the production of a crystallinity degree between 40 and 70 %. Among this material properties, low density, high stiffness, high surface hardness, high chemical resistance and extremely low water absorption stand out (favorable factor for use in water treatment system) [7].

*N*-hydroxymethyl acrylamide (HMAA), or *n*-methylol acrylamide (NMA), is a water-soluble monomer widely used for preparing crosslinked copolymers by emulsion and solution polymerization [8-11]. The methylol groups do not react with free radical polymerization initiators, but readily condense themselves by heat curing producing networks (heating promotes condensation reactions between the hydroxyl groups of the methylol ether producing intermolecular bonds). The temperature control is essential to control the crosslinking degree. The heating above 373 K may lead to ether links breakage, with methylene bridges production [11]. The initiator chemical nature and the reaction medium affect the crosslinking degree: benzoyl peroxide and 403 K temperature promote a high crosslinking degree while the initiation of potassium persulfate leads to a gel production which water removal and network formation can be controlled by heating after reaction [12-13]. The HMAA is used in fabrics, adhesives, elastomers, and fibers

industry, but, despite its wide application, there are few studies related to crosslinking HMAA reactions [5]. The resulting crosslinked polymers are water insoluble, heat stable, and have varying degrees of porosity depending on the crosslinking degree [14]. The porosity increase contributes to the increase of surface area, favoring the adsorption of larger molecules present in most industrial waters. Due to these properties, it is used for several purposes [15-21].

In this work, polymer composites were obtained from the reaction by free radical polymerization of HMAA in the presence of post-consumer PP with subsequent condensation reaction catalyzed by heating. The products were characterized by Fourier transform infrared spectrometry (FT-IR), optical microscopy (OM), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and X-ray diffraction (XRD). Moreover, the bulk density and the degree of swelling were also determined.

## 2. Experimental

### 2.1. Materials

*N*-hydroxymethyl acrylamide (HMAA) 48 % (w/v) aqueous solution was supplied by Aldrich (Sao Paulo). Post-consumer packaging margarine Qualy®, produced with polypropylene homopolymer (HP502H and JE6100) were used after detergent cleansing followed by alcohol. Ethanol (95 %) was supplied by Quimex (Sao Paulo). Hexane PA and potassium persulfate 99 % were provided by Vetec Quimica Fina Ltda (Rio de Janeiro). Carbon tetrachloride PA was supplied by Rio Lab (Goiania). All materials were used as received.

### 2.2. Synthesis of PP/PHMAA Compounds

#### Preparation of PP support

The post-consumer packages have been made at around 2x6 mm size and powdered with the aid of an analytical Quimis mill. Grinding was done under cooling with water at 288 K. The resulting powder was sieved with a 40 mesh Retsch.

#### Synthesis of composite polypropylene/poly (hydroxymethylacrylamide) (PP/PHMAA)

The polymer blends were prepared by monomer reaction in the presence of post-consumer PP. The PP/HMAA proportions were 0.40, 1.48 and 2.66 by mass; the resulting compounds were named P01, P02 and P03, respectively. All reactions were carried out at HMAA constant mass varying PP mass. Therefore, it was possible to evaluate the influence of increasing concentrations of PP on the properties of the polymer composite. The synthesis

was carried out in a three necked round bottom flask with magnetic stirring, by adding 10 ml of 50 % aqueous ethanol, 3 ml dry ethanol, 2.7 ml aqueous HMAA (48 % w/v), and PP powder. At 343 K, 2 ml K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> were added to the flask. After around 15 min, a change was observed from liquid to gel, where the temperature raised to 353 K and the reaction went on for other 10 min. At this point the cross linking was achieved. After that, the flask was rapidly cooled at ice bath and the contents was washed with water using conical flask and funnel under vacuum. The wet gel, disposed on a Petri dish, was dried at 323 K for 48 h and after that the product was hard and dry. The sample was then slightly moistened with distilled water, grounded with the aid of level and pestle, and dried at 323 K for 24 h. The product was kept free of moisture in a desiccator.

#### Homogenization of grain size of the compound PP/PHMAA

In order to obtain more uniform particle size materials, the product recovered after passage through Retsch sieve of 25 mesh Retsch sieve.

### 2.3. Characterization of Compounds

PP/PHMAA compounds after crosslinking reaction were characterized by Fourier transform infrared spectroscopy (FT-IR), optical microscopy (OM), differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA), and X-ray diffraction (XRD). Moreover, the bulk density and the degree of swelling were also determined.

#### Absorption spectrometry, infrared (FT-IR)

Analyses were performed using an absorption spectrophotometer in the infrared region with Perkin Elmer Spectrum BX and 1720-X FTIR. Absorption spectra, in the transmission mode, were obtained from the compound and KBr pressed as disks at 1 mg/100 mg ratio. The composition of each sample was calculated using an absorbance ratio response curve  $A(1559 \text{ cm}^{-1})/A(1377 \text{ cm}^{-1})$  as function of PP/PHMAA proportion. This curve was built from absorbance values obtained for known compositions mixtures of PP and HMAA homopolymers. The absorption at  $1377 \text{ cm}^{-1}$  is attributed to the angular deformation of CH<sub>3</sub> groups of PP and the absorption at  $1559 \text{ cm}^{-1}$  is due to CN and NH of PHMAA (amide II band) [22].

#### Bulk density

A graduate cylinder was filled with PP/PHMMA compound until a fully dense bed with constant volume was obtained; the cylinder was filled up with the aid of an IKA Vibro-fix tube stirrer. The apparent density ( $d_{\text{bulk}}$ , g/ml) was obtained from the polymer mass ( $M_a$ , g) and volume ( $V$ , ml) ratio was read in the cylinder, as shown in Eq. (1):

$$d_{\text{bulk}} = \frac{M_a}{V} \quad (1)$$

Swelling volumetric percentage

For percentage swelling volume analysis ( $I$ ), or swelling degree, the three compounds, P01, P02 and P03, were placed in 10 ml beakers at 0.3750, 0.2991 and 0.2229 g, respectively. These compounds occupy a volume of approximately 1.0 ml ( $V_i$ ). Subsequently, water was added in an amount equivalent to two and a half times the volume occupied by the dry polymer (approximately 2.5 ml), leaving at rest for 24 h. After this period, it was assumed that polymers reached the equilibrium swelling and the volume of the swollen polymer ( $V_f$ ) was measured again. The swelling percentage was calculated using Eq. (2). This test was performed in triplicate.

$$I = \frac{(V_f - V_i)}{V_i} \cdot 100 \quad (2)$$

The crosslinking degree ( $X_c$ ) can be calculated from the equilibrium swelling data using Eq. (3) [23-24], observing that  $X_c = M_m/M_x$ ,  $M_m$  being the molecular mass of monomeric units and  $M_x$  the molecular mass per PHMAA crosslinking unit:

$$M_x = \frac{-V_1 \cdot r_2 \cdot (c^{1/3} - c/2)}{\ln(1-c) + c \cdot c^2} \quad (3)$$

where  $V_1$  – a solvent molar volume, 18.06 ml/mol (at 298 K) [25];  $\rho_2$  – PHMAA density, 1.2194 g/ml [26];  $c$  – Flory interaction parameter for PHMAA-water;  $c$  – swollen polymer volume fraction at equilibrium ( $c = M \cdot V_f / \rho_2$ );  $V_f$  – swollen polymer volume;  $M$  – polymer initial mass.

Optical microscopy

The grain morphology, such as sphericity and opacity, was evaluated with the aid of a BX50 Olympus optical microscope with video camera coupled to a microcomputer. The samples were deposited on glass slides and observed in transmission mode with analyzer and crossed polarizer and analyzer.

Differential scanning calorimetry (DSC)

The DSC analyzes were conducted on a DSCQ-1000 TA Instruments, from 303 to 473 K at 10 K/min under 60 ml/min constant nitrogen flow. The amount of material used for analysis was approximately 5.0 mg. Data were taken from the curves obtained for the second heating procedure.

Thermogravimetric analysis (TGA)

TGA analyzes were performed on a TGA 951 TA Instruments from 303 to 473 K at 10 K/min under 60 ml/min constant nitrogen flow. The amount of material used for analysis was approximately 5.0 mg.

X-ray diffraction (XDR)

The crystallinity degree was determined from the analysis at a XDR-6000 Shimadzu with Copper  $K\alpha$

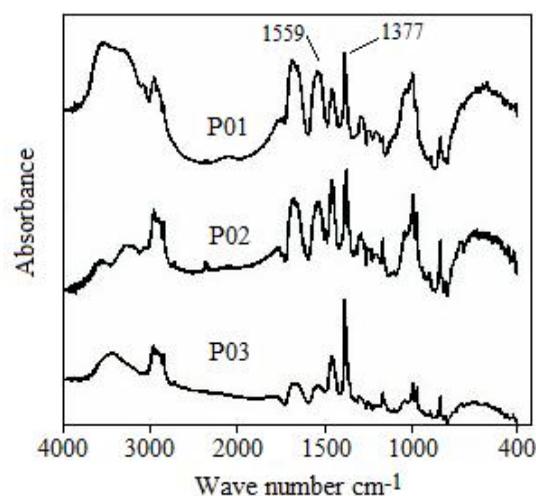
radiation, using powder method. A sample free of moisture was pressed on the specimen holder. The speed was 2 $\theta$ /min and 2 $\theta$  varied from 5.0 to 80.00 degrees.

### 3. Results and Discussion

The synthesis performed in this study to obtain a crosslinked polymer composite did not use crosslinking agents. The *n*-hydroxymethyl acrylamide (HMAA) was polymerized with potassium persulfate in the presence of powered PP; after curing, through heating, condensation reactions that promoted crosslinking occurred in the system. This procedure facilitated the final product grinding, which was solid, infusible and insoluble.

#### 3.1. Absorption Spectroscopy in the Infrared Region (FT-IR)

From products FT-IR spectra (Fig. 1) and response curve ( $y = 0.0349x - 0.4703$ ;  $R_2 = 0.9877$ ) equation [27], the compositions of the compounds P01, P02 and P03 was determined, which are presented in Table 1. It is observed that the levels of obtained PHMAA 35, 30 and 21 wt % for P01, P02 and P03, respectively, are far below the ones fed to the reactor: 71.4, 40.3 and 27.3, respectively, indicating a relatively low conversion of PHMAA in the presence of grounded PP.



**Fig. 1.** Infrared spectra of crosslinked PP/PHMAA supports corresponding to the feed composition given in Table 1

In order to verify the reproducibility of the synthesis, the reaction related to PP/PHMMA 28.6:71.4 (w/w) was done in triplicate producing blends presenting 35.5, 36.1 and 37.3 wt % of PHMMA. This corresponds to 1 % standard deviation.

Table 1

**P01, P02 and P03 composition obtained from the ratio  $A(1559\text{ cm}^{-1})/A(1377\text{ cm}^{-1})$   
and from the response curve equation:  $y = 0.0349x - 0.4703$**

Support	$A(1559\text{ cm}^{-1})$	$A(1377\text{ cm}^{-1})$	$A(1559)/A(1377)$	PHMAA, wt %
P01	0.02888	0.03880	0.7443	35
P02	0.01820	0.03050	0.5967	30
P03	0.01690	0.06489	0.2604	21

Note:  $y = A(1559)/A(1377)$  and  $x = \text{wt \% of PHMAA}$

Table 2

### Bulk density of PP/PHMAA composites

Support	Weight composition PP:PHMAA	$d_{\text{bulks}}$ g/ml	Swelling, %	$X_c$
P01	65:35	0.37	30	$0.9737 - 0.0560c$
P02	70:30	0.30	80	$0.3344 - 0.0185\chi$
P03	79:21	0.22	100	$0.1594 - 0.0084\chi$

Note:  $c = \text{Flory interaction parameter } (< 0.5)$

## 3.2. Determination of Bulk Density, Percentage Swelling and Crosslinking Degree

Table 2 shows the bulk density results ( $d_{\text{bulk}}$ ), the swelling percentage ( $I$ , %) and the equation for calculating the crosslinking degree ( $X_c$ ) for P01, P02 and P03. The crosslinking degree is expressed in terms of Flory interaction parameter ( $c$ ) which value is less than 0.5; therefore, it is possible to conclude that the compounds crosslinking degree increases in the following order: P03 < P02 < P01.

As expected, the bulk density increases with increasing PHMAA content, once this polymer density (1.2194 g/ml) [26] is greater than the polypropylene density (0.90 g/ml) [28]. However, the compounds density values obtained are far below their respective pure homopolymers reported in the literature [26, 28], indicating the production of materials with high contents of voids. Such high porosity can be observed for the compounds swelling degree: P03 with the lowest bulk density showed a swelling degree in water of 100 %.

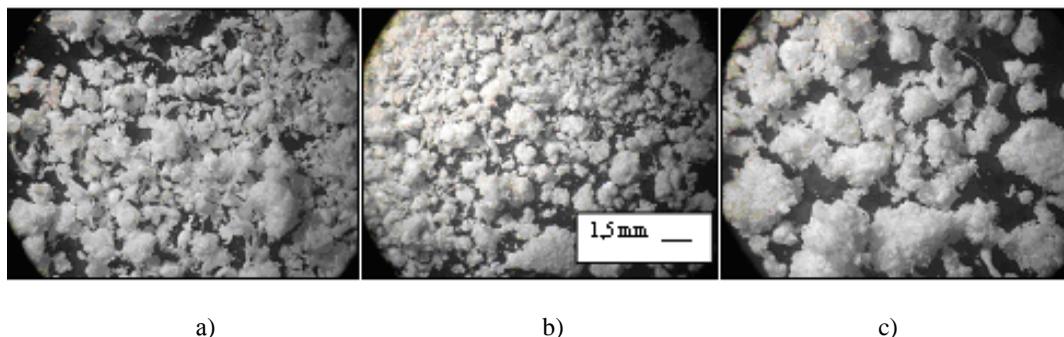
## 3.3. Optical Microscopy

Figs. 2 and 3 show the micrographs from an optical microscope for moistened and dried P01, P02 and P03, respectively. For dry samples (Fig. 2), it can be seen that the size of the particle aggregates increases with increasing PP. P03 seems spongier than P02 and P01. This is better seen in the wet samples (Fig. 3). Once moistened, the P03 volume increases more than that of P02 and P01 supports, as already evidenced by the results presented in Table 2. For P01, which swells less when in contact with

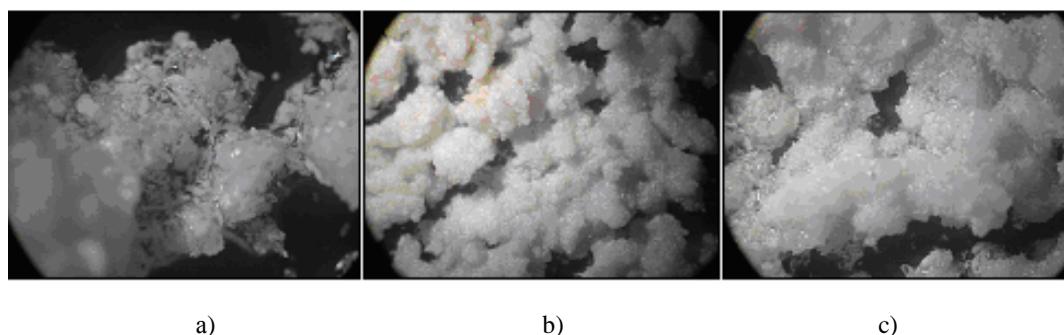
water, the presence of smooth and compact PHMAA structures is observed. Once this compound has a higher polar groups content (OH, C=O and NH) than methylolacrylamide and PP is a nonpolar polymer, one may expect that P01 support swelled more in water than the other compounds. However, this does not happen because P01 has a higher crosslinking degree with more dense and closed morphological structure, which restricts the swelling. It is considered that the PP chains (nonpolar) retained between the polar PHMAA chains cause repulsion of PHMAA chains, leading to the spongy structure and lower crosslinking degree observed for P03 when compared to P01 and P02.

## 3.4. Thermogravimetric Analysis (TGA)

Table 3 summarizes data extracted from the percentage mass loss curves as a function of increasing temperature for PP, PHMAA, P01, P02 and P03. Non-crosslinked PHMAA lost 14.3 % of its mass when heated from room temperature to 423 K. This mass loss is attributed to solvent loss. On the other hand, PP is thermally stable up to 423 K, not exhibiting any mass loss up to that temperature. The behavior of P01, P02 and P03 up to 423 K agrees with their composition, that is, the amount of mass loss increases with PHMAA contents increase. As observed in optical microscopy analysis, P01 support (35 % PHMAA) absorbs less water than P02 (30 % PHMAA) and P03 (21 % of PHMAA), despite their higher PHMAA concentration. This observation indicates that the largest amount of volatiles (water and aldehyde) comes from condensation reactions; absorbed solvents (water and ethanol) contribute to a lesser extent.



**Fig. 2.** Optical microscope micrographs for dry compounds: P01 (35 % PHMAA) (a); P02 (30 % PHMAA) (b) and P03 (21 % PHMAA) (c)



**Fig. 3.** Optical microscope micrographs for compounds moistened with water: P01 (35 % PHMAA) (a); P02 (30 % PHMAA) (b) and P03 (21 % PHMAA) (c)

*Table 3*

**Data obtained from percentage weight loss curves as a function of temperature increase: % mass loss up to 423 K (mass loss < 423 K); % mass loss up to 573 K (mass loss < 573 K); temperature at which there is maximum degradation rate ( $T_{max}$ ); number of degradation stages taken from the derivative curve (# stages)**

Material	mass loss < 423 K, %	mass loss < 573 K, %	$T_{max}$ , K	# stages
PHMAA	14.3	25.0	343.0	3
PP	0.0	4.0	661.5	1
P01 (PP/PHMAA 65/35)	9.0	24.0	654.2	5
P02 (PP/PHMAA 70/30)	5.4	18.0	668.5	4
P03 (PP/PHMAA 79/21)	2.0	10.0	663.5	3

Regarding the PP degradation profile, as expected, it shows a single degradation stage with maximum weight loss speed at 661 K. P01, P02 and P03, however, have more than one degradation stage. The number of stages increases with increasing PHMAA contents: P03 (21 % PHMAA) has three maxima at 474, 620 and 651 K (Fig. 4); P02 (30 % PHMAA) has four stages of degradation (474, 594, 633 and 668 K); and P01 (35 % PHMAA) has five stages of degradation (474, 563, 593, 634 and 654 K). Once PP, at 573 K has lost 4 % of its mass (Table 3) and the maximum peak at 620 (P03), 594 (P02) and 593 K (P01), the degradation at higher temperatures is related to the beginning of PP degradation along with PHMAA degradation.

### 3.5. Differential Scanning Calorimetry (DSC)

The heat flow vs. temperature curve for PP shows, as expected, only one endothermic peak, at around 438 K, characteristic of the material melting [7]. On the other hand, the PHMAA curve shows two exothermic peaks, one at 392 K and another – at around 479 K. These peaks are related to the material degradation since this material does not melt [26]. In the curves obtained for P01, P02 and P03, in the first heating run, two endothermic peaks are observed attributed to PP melting and to PHMAA degradation.

This degradation, confirmed in the second heating in which only the PP peak is observed, was also observed in the literature [27]. It is attributed not only to the volatilization of water and ethanol solvents used in the synthesis but also to condensation reaction of HMAA OH groups producing ether bridges, followed by methylene bridges, releasing water and aldehyde, respectively [29]. Fig. 5 shows the heat flow curves as a function of heating for P01. The curves obtained for P02 and P03 are similar to those displayed in Fig. 4, with the exception of the peak areas related to PHMAA degradation which decreases in the following order: P01 > P02 > P03. The DSC endotherms areas are proportional to the percentage weight lost: 9.0, 5.4 and 2 wt % for P01, P02 and P03, respectively (Table 3).

Table 4 summarizes the temperature values corresponding to the endotherms, at maximum peak, for P01, P02 and P03 and the melting enthalpy for pure PP. The results of the first run show that by increasing PHMAA mass in the polymer, the temperature at the maximum of the first DSC endotherm curve also increases, getting closer to the temperature observed for pure PHMAA (391.9 K). It also was observed in the first run that PHMAA had no significant effect over the PP melting temperature. Conversely, in the second run, it was observed that this melting temperature decreased by 2.0 K (P01, 35 % PHMAA) and 2.7 K (P02, 30 % PHMAA) for compounds containing 35 and 30 % PHMAA, respectively; however, 21 % PHMAA for P03 had no effect over the PP melting temperature. The lowering of the melting point of crystalline polymers due to the addition of an amorphous polymer component is predicted by the Flory-Huggins equation and occurs even in the case of incompatible polymers blends. It is attributed to the effect of the amorphous polymer in the morphology modification, reduction of crystal size and introducing defects in the crystal lattice [30].

Regarding PP fusion enthalpy observed in first heating, it increases with increasing the PP contents in

PP/PHMAA composite, causing an increase in PP melting temperature. This behavior shows that PHMAA synthesis in PP presence interfered with the lamella thickness of PP molecules (higher melting temperature), indicating the existence of physical interactions between polymers chains. It is also noted that in PP/PHMAA samples the degree of crystallinity of PP is lower than that in pure PP, demonstrated by the lower fusion enthalpy. The interaction of PP with PHMAA is also confirmed by the increase of PP fusion enthalpy as increasing PP content in the composite, as observed in the second heating, shown in the third column of the second run. The difference between the PP melting temperatures for the polymers in the first heating when compared to the second heating increases with increasing concentration of PHMAA, *i.e.* 0.5, 2.3 and 2.8 K, for 21, 30 and 35 % PHMAA, respectively. In all compounds, it was observed in the second heating that the endotherm occurred at a wider temperature range than the first heating. This shows that the PP crystals formed during the molten material cooling have a broader size distribution, with a high incidence of smaller crystals than crystals in the first run. This does not happen with pure PP.

The melting enthalpy for PP in the compounds, in the second heating, when compared to the first heating, increased by 17.6 and 14.5 J/g for P03 and P02, respectively, while the melting enthalpy of post-consumer PP increased by only 2.5 J/g. The initial melting temperatures, which could be associated to the initial process of crystallization under cooling, increases as increasing PHMAA content in the polymer blend, that is, the presence of PHMAA results in the crystal formation in advance as compared with pure PP. All these results together suggest that larger amount of crystals is formed in the compounds than for post-consumer PP; therefore, one may conclude that PHMAA acts as a nucleating agent for PP crystallization from the molten one. This is expected once PP crystallizes by heterogeneous nucleation.

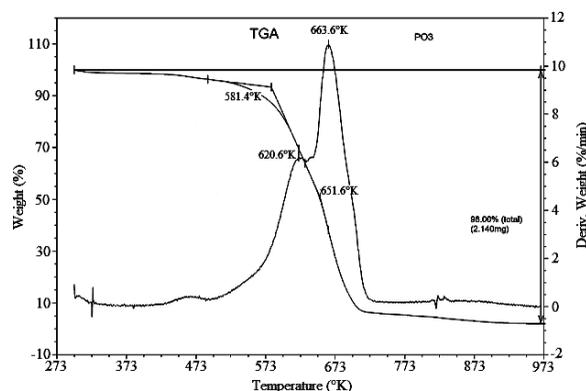


Fig. 4. TGA and DTG curves for P03 (PP/PHMAA 79/21)

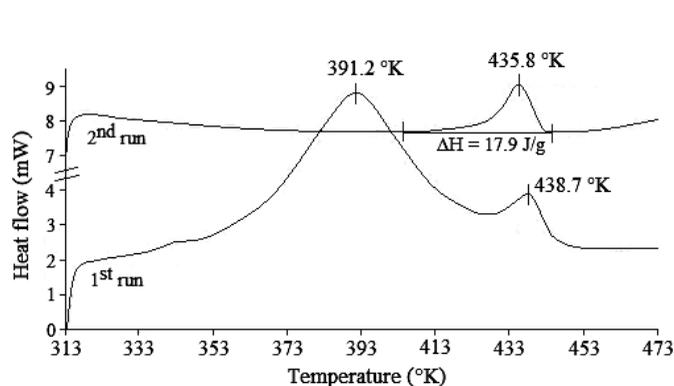


Fig. 5. DSC curves for P01 (PP/PHMAA 65/35) obtained at the first and second heating

Table 4

Temperatures at peak maximum for PP fusion and PP/PHMAA, as well as PP enthalpies

Sample	First heating				Second heating		
	Temp. at PHMAA peak, K	Temp. of PP melting, K	Range of melting, K	Enthalpy of PP melting**, J/g	Temp. of PP melting, K	Range of melting, K	Enthalpy of PP melting**, J/g
PP *	–	440.2	409-453	80.2	438.6	402-450	82.7
PHMAA	391.9	–	–	–	–	–	–
P01 PP/PHMAA 65/35	391.2	438.6	426-448	***	435.8	411-443	27.6
P02 PP/PHMAA 70/30	371.2	438.8	422-449	44.9	436.6	407-446	59.4
P03 PP/PHMAA 79/21	356.2	440.6	419-451	63.3**	440.2	404-449	74.9

Notes: \*Post-consumer PP. \*\*Values already considering PP concentration in the samples. \*\*\*Not determined.

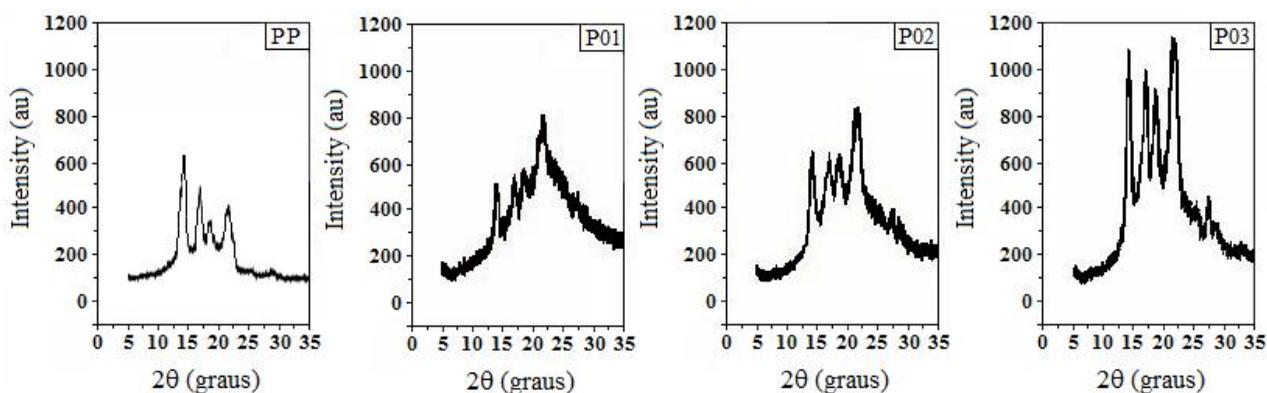


Fig. 6. X-ray diffractograms for PP, P01 (PHMAA 35 %), P02 (30 % PHMAA) and P03 (21 % PHMAA)

### 3.6. X-ray Diffraction (XRD)

X-ray diffractograms for PP, P01, P02, and P03 are given in Fig. 6. One may observe the crystalline peaks attributed to isotactic PP in  $\alpha$ form, at  $2\theta$  of 14.1; 16.9; 18.6; 21.2 and 21.8 degrees [7] and a halo which corresponds to amorphous PP and PHMAA. The crystalline area related to the halo peaks increases with increasing the proportion of PP in the sample, as expected. The intensity of the peaks at  $2\theta$  equals to 21.2 and 21.8 degrees regarding the peak at 14.1 degrees decreases with increasing the PP concentration, showing possible distortions in the crystal lattice. This shows that PP crystallization was affected during HMAA polymerization, as also been suggested for DSC analysis.

### 4. Conclusions

The polymerization reaction of *n*-hydroxymethyl acrylamide (HMAA) in the presence of polypropylene (PP) leads to a cross-linked structure producing network polymer blends of PP and PHMAA; the product is an insoluble and infusible solid. The reactions were reproducible in terms of PP/PHMAA composition with 1 % standard deviation. The particle size increases with increasing PP contents as well as the swelling capacity: P03 with high PP concentration (79 %) presents spongier and swells 3.3 and 1.5 times more than P02 (70 % PP) and P01 (65 % of PP) supports, respectively. This behavior is associated to the crosslinking degree of the compounds which decreases in the following order: P01 > P02 > P03. The synthesis of PHMAA in the

presence of PP interferes the crystal habit of PP molecules, indicating the presence of physical interactions between the chains of these polymers. The following aspects were also observed: i) decrease in the melting enthalpy of PP in the compound when compared to pure PP; ii) reduction of the melting temperature for PP in the compounds with PHMAA concentration increase; and iii) a change in X-ray diffraction. The thermal stability of the compounds was higher for higher levels of PP. Thus, polymer blend network with high levels of post-consumer material shows the required characteristics for water treatment, especially if containing organic contaminants.

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## References

- [1] Paranhos C., Oliveira M., Soares B. and Gomes A.: Polímeros, 2009, **19**, 196.
- [2] Celestino M. and Soares B.: Polímeros, 2013, **23**, 139.
- [3] Lopez-Gil A., Rodriguez-Perez M., De Saja J. et al.: Polímeros, 2014, **24**, 36.
- [4] Ramoa S., Merlini C., Barra G. And Soares B.: Polímeros, 2014, **24**, 57.
- [5] Olabisi O., Roberson L. and Shaw M.: Polymer-Polymer Miscibility. Academic Press, New York 1979.
- [6] Paul D., Barlow J. and Keskula H.: Polymer Blends [in] Mark H., Bikales N., Overberger C. and Menges G. (Eds.), Encyclopedia of Polymer Science and Engineering. John Wiley & Sons, New York 1987, v.12.
- [7] Moore E.: Polypropylene Handbook. Hanser Publishers, Munich 1996.
- [8] Bufkin B. and Grawe J.: J. Coat. Technol., 1978, **50**, 41.
- [9] Zosel A. and Ley G.: Macromolecules, 1993, **26**, 2222.
- [10] Volfova P., Chrastova V., Cernakova L. et al.: J. Macromol. Symp., 2001, **170**, 283.
- [11] Kamogawa H. and Sekiya T.: J. Polym. Sci. A., 2013, **50**, 2011.
- [12] Rosamilia P. and Davidowich G.: USA Patent 4590102A, Publ. May 20, 1986.
- [13] Krishnan S.: PhD. thesis, Lehigh University, Bethlehem USA, 2002.
- [14] Klempner D. and Berkowski L.: Interpenetrating Polymer Networks [in] Mark H., Bikales N., Overberger C. and Menges G. (Eds.), Encyclopedia of Polymer Science and Engineering. John Wiley & Sons, New York 1987, v.8.
- [15] Coutinho F., Rezende S. and Barbosa C.: Polimeros, 1999, **10**, 129.
- [16] Marquardt M. and Eifler-Lima V.: Quim. Nova, 2001, **24**, 846.
- [17] Entry J., Sojka R., Watwood M. and Ross C.: Environ. Pollut., 2002, **120**, 191.
- [18] Mansour E. and Dawoud F.: J. Sci. Food Agric., 2003, **83**, 446.
- [19] Dalla-Vecchia R., Nascimento M. and Soldi V.: Quim. Nova, 2004, **27**, 623.
- [20] Bajpai S. and Johnson S.: React. Funct. Polym., 2005, **62**, 271.
- [21] Queiros Y., Clarisse M., Oliveira R. et al.: Polimeros, 2006, **16**, 224.
- [22] Silverstein R. and Webster F.: Spectrometric Identification of Organic Compounds, John Wiley & Sons, New York 2005.
- [23] Flory P.: J. Chem. Phys., 1950, **18**, 108.
- [24] Barikani M. and Hepburn C.: Iran. J. Polym. Sci. Technol. 1992, **1**, 1.
- [25] Lide D.: Handbook of Chemistry and Physics. CRC Press, Florida 2001.
- [26] Watanabe D. and Esperidiao M.: Annals XVIII Congresso Brasileiro de Engenharia Química, Brazil, Campos de Jordão 2010.
- [27] Esperidiao M. and Rocha L.: Annals 22° Reuniao Anual da SBQ, Brazil, Pocos de Caldas 1999.
- [28] Brandrup J. and Immergut E.: Polymer Handbook, 3<sup>rd</sup> edn. John Wiley & Sons, New York 1989.
- [29] Marinova K., Alargova R., Denkov N. et al.: Langmuir, 1996, **12**, 2045.
- [30] MacKnight W., Karasz F. and Fried J.: Solid State Transition Behavior of Blends [in] Paul D. and Seymour Newman (Edn.), Polymer Blends. Academic Press, New York 1997, 186-238.

## ОДЕРЖАННЯ ТА ХАРАКТЕРИСТИКА СПОЛУК НА ОСНОВІ ПОЛІ(ГІДРОКСИМЕТИЛ АКРИЛАМІДУ) І ПОСТ-СПОЖИВЧОГО ПОЛІПРОПІЛЕНУ

**Анотація.** З метою отримання матеріалів для оброблення стічних вод нафтової промисловості синтезовані полімерні композити. Синтез проводили реакцією полімеризації за участю вільних радикалів з використанням н-гідроксиметил акриламід у присутності пост-споживчого поліпропілену (ПП) з подальшою реакцією конденсації за нагрівання, що дозволяє уникнути використання структуруючих агентів. Характеристику продуктів проведено за допомогою Фур'є ІЧ-спектроскопії, оптичної мікроскопії, диференціальної скануючої калориметрії, термогравіметричного аналізу і рентгенівської дифракції. Визначено насипну густина та ступінь набухання продуктів. Показано, що синтез є відтворювальним, а використання ПП від упакування харчових продуктів знижує вартість виробництва. Здатність до набухання і термостійкість композиту зростає зі збільшенням вмісту ПП в суміші.

**Ключові слова:** полімерний композит, полімерна суміш, пост-споживчий поліпропілен, полі(гідроксиметил акриламід), реакція структуривання, характеристика полімеру.