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Synthesis and Absorption Study of Polystyrene-*graft*-Poly (acrylic acid)/ Hectorite Superabsorbent Polymer Composite

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ABSTRACT

A novel of Superabsorbent Polymer Composite (SAPC) was synthesized through emulsion polymerization technique based on partially neutralized acrylic acid (AA) grafted onto waste polystyrene foam using N,N'-Methylenebisacrylamide (NMBA) and Ammonium Persulphate (APS) , as crosslinker and initiator respectively. The purpose of this study is to prepare a superabsorbent polymer hydrogel composite using hectorite clay as the reinforcement and also to study the effect of incorporation of hectorite clay on water uptake capacity .Swelling studies were carried out in distilled water and saline water. Besides, SAPC was characterized using Fourier Transform Infra Red (FTIR) .The FTIR results confirmed that the grafting polymerization took place among the polystyrene, acrylic acid and hectorite clay. Then, the introduction of hectorite particles in the superabsorbent polymer increased the water absorbency.Superabsorbent composite containing 6 wt % hectorite has the highest water absorbency (65.34 g/g in distilled water and 21.3 g/g in 1.0 wt % NaCl solution).

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INTRODUCTION

Superabsorbent polymer (SAP) or is known as hydrogel is a three dimensional crosslinked polymer networks which have a great ability in absorbing huge amount of liquid and retaining the liquid within their structure without dissolving it (Buccholz, 1998) . Superabsorbent polymer is a hydrophilic polymer in which it can absorb water from 10% up to thousands of their own weight in a very short period of times (Osada, 2001). Hydrophilicity effect in this hydrogel is attributed by the presence of polar groups, which can be non-ionic (-OH, -O-,-NH2, -CONH-,-CHO) or ionic (-SO3H,-COOH,-COONa,-COONH4) (Zhang, 2007). Besides, water absorbed by superabsorbent polymers is hardly removed although some pressure is applied to it (Dafader, 2009). With all of these excellent properties, SAPs are widely used in wide range of applications such as hygienic product, diapers, medicine, cosmetics and agriculture (Karadag, 2002). Lately, waste polystyrene foam has become a critical environmental issue as it is one of the sources of environmental pollution. Polystyrene is a synthetic hydrophobic non biodegradable polymer which takes a very long time to be decomposed in nature. Once it is disposed, it will take up spaces and landfills and cause environmental pollution. There are different alternatives in order to reduce this problem and convert it into valuable product, including polymer modification and functionalization. Utilization of waste polystyrene foam in order to produce new materials is a kind of polymer waste management which is crucial to our environment. The introduction of polar groups into synthetic polymer caused the induction of hydrophilicity (El, 2013). In recent years, studies had been made to increase the strength of superabsorbent polymers. This is because they have low gel strength which restrict their applications. To improve these limitations, inorganic mineral clay such as Montmorillonite, Kaolin and Bentonite has been used in the preparation of superabsorbent polymer composite by introducing the clays into organic polymer networks. The clay used in this work is Hectorite. Hectorite clay is a rare magnesium lithium clay cations belongs to smectite clay mineral group having reactive –OH group on its surface which enable the absorption of water inside them (Sadeghi,

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2013). Hectorite is characterized by a negatively charged plate-like structure with exchangeable associated cation counterion sandwiched between plate-like layers. During swelling, water molecules penetrate into the interlayer spacing of hectorite, then the Na cations dissociate as they hydrate allowing the negatively charged platelets to repel each other and give them their unique swelling properties (Robert, 1996).

Sheng *et al* synthesized a superabsorbent polymer composite using waste polystyrene foam, acrylic acid and Montmorillonite through emulsion polymerization technique (Sheng Liu, 2007, 2006) An Feng Zhu *et al* prepared a superabsorbent polymer composite via in situ free radical polymerization using hectorite clay as crosslinker and Ammonium Persulphate as initiator in order to increase the hydrogel strength and swelling properties (Zhu, 2013). The novelty in this project is the incorporation of hectorite clay as the reinforcement since no work and research has been performed using these clay mineral.

Methodology:

Materials:

Acrylic Acid (AA, stabilized with hydroquinone monomethyl ether for synthesis) was purchased from Merck. Toluene and ethyl acetate were obtained from Scharlau. n-Hexane was obtained from Laboratory Chemicals. N,N-Methylenebisacrylamide(NMBA),Ammonium Persulphate (APS) and Span 60 were purchased from Sigma Aldrich. Sodium hydroxide (NaOH, extra pure) was supplied by Loba Chemie. Waste polystyrene foam (WPSF) obtained from food container was washed with distilled water and dried at room temperature before use. Sodium type Hectorite (Na-Hec) was modified from original Hectorite (supplied by Guangzhou Bofeng Chemical Technology Co.,Ltd) in our lab..

Instrumental Analysis:

FTIR spectra of the prepared samples were performed using Perkin Elmer Spectrum One apparatus operating in the range of 4000 to 450cm-1. The dried samples were grinded with KBr powder and pressed into tablets before spectrum acquisition.

Preparation of Na-Hectorite:

The Na-Hectorite was prepared by stirring 5g of the sample in 500mL of 1.0*M* NaCl solution for 24 hours. The Na-Hectorite was separated from the excess solution by centrifugation and washed several times to remove the Cl-. Then, the product was dried at a temperature of 105^{o}C over night. Na- Hectorite powder was obtained by grinding the dried samples using mortar and pestle.

Preparation of Polystyrene-graft-Poly(Acrylic Acid)/ Hectorite superabsorbent polymer nanocomposite:

The synthesis of superabsorbent polymer composite began with the preparation of the first solution in which waste polystyrene foam (WPSF) was dissolved in a mixed solvent consists of n-Hexane, toluene and ethyl acetate in the ratio of 3:8:6. Then, certain amount of (Span 60) dispersant was added in the solution under stirring. In another beaker, second solution was prepared.Acrylic acid (AA) was partially neutralized with 10% sodium hydroxide (NaOH) solution in an ice bath and then a proper amount of Na-Hectorite was added to the acrylic acid solution under stirring for a few minutes. Both of these solutions were then transferred to three neck reaction flask equipped with thermometer, a mechanical stirrer and nitrogen line. The solution was heated to45^{o}C and then to 70^{o}C. When temperature reached 70^{o}C, an appropriate amount of initiator (APS) and crosslinker (NMBA) was added and stirred until it reached the gel point. Once it became gel, the temperature was held at 70^{o}C for two hours without stirring. Then, the gel was precipitated into acetone in order to remove the unreacted monomer, cut into small pieces and dried in an oven at 100^{o}C for 24 hours.

Preparation of PS-g-PAA superabsorbent polymer:

The preparation method of PS-g-PAA superabsorbent polymer used was similar to that of the PS-g-PAA/HT superabsorbent polymer nanocomposite except that Hectorite was not included.

Water absorbency measurement:

A predetermined amount of dried sample (1g) with particle size between 60 and 100 mesh was immersed in 100mL of distilled water and saline solution (1 wt %) , and the samples were allowed to soak at room temperature to reach the swelling equilibrium for 2 h. Then, the excess water was removed by filtrating the samples using screen and the samples were weighed. The swelling absorbency (A) of the samples were calculated using the following formula (Sheng,2006):

$$A = (m2 - m1)/m1 \quad (1)$$

Where A is water absorbency, *m2* is the weight of swelling sample and *m1* is the weight of dried samples.

Results:

Fourier Transform Infra Red (FTIR):

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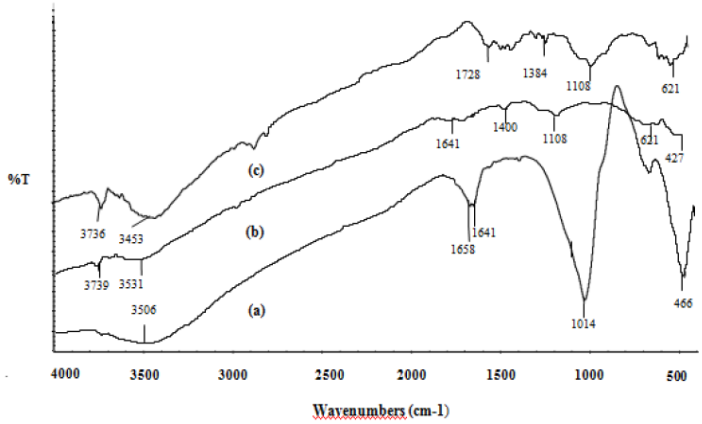


Fig. 1: FTIR spectra of (a) hectorite, (b) PS-g-PAA, and (c) PS-g-PAA/HT.

Water Absorbency Test

Table 1: Effect of hectorite loading on water absorbencies of superabsorbent polymer composite.

Sample	Hectorite Percentage(wt%)	A (g/g)	
		Distilled water	NaCl (1.0 wt%)
PS-*g*-PAA/HT$_0$	0	33.83	9.52
PS-*g*-PAA/HT$_1$	2%	39.8	15.3
PS-*g*-PAA/HT$_2$	4%	44.9	15.2
PS-*g*-PAA/HT$_3$	6%	65.34	21.3
PS-*g*-PAA/HT$_4$	8%	47.35	13.04

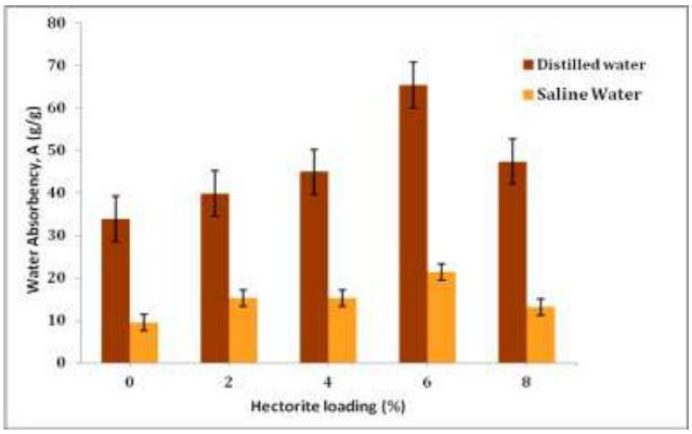


Fig. 2: Water absorbency of PS-g-PAA/HT in distilled water and saline water.

Discussion:

FTIR spectrum Analysis:

The FTIR of hectorite**,** PS-g-PAA and PS-g-PAA/HT are shown in Figure 1. In the spectrum of hectorite, the broad band at 3506cm-1 arises due to the stretching band of the O-H groups on the surface of the clay. The bands at 1658cm-1 and 1641 cm-1 are related to the first overtone of the O-H bending vibration. The band at 1014cm -1 is related to Si-O vibration and 466cm -1 corresponds to the Si-O-Si bending vibration respectively (Ismail, 2012).In PS-g-PAA spectra, the sharp band at 3739cm -1 and broad band at 3531cm-1 correspond to the O-H stretching vibration from hectorite clay and acrylic acid. The peak at 1641cm -1 is due to the C=C bending of the vinyl group from PS and C=O stretch of amide group from the crosslinker while the band at 1400cm -1 results from C-H bending and deformational vibration of –CH2 of PS respectively. Additionally, the band at 1108 cm -1 is related to C-O stretch of 1728cm-1 is related to C=O stretching band of acrylic acid. After hectorite was introduced into the polymeric network, the absorption band of –CH2 and –COO- shifted from 1400cm-1 to 1384cm-1 .Besides, the absorption band at 1384cm-1 is also related to S=O group from initiator used. In addition, the absorption band at 1108cm-1 is due to C-O stretch while the peak at 621cm-1 due to C-H deformation. From figure 1, it can be concluded that

grafting polymerization took place among the polystyrene, acrylic acid and hectorite.

Influence of Hectorite clay loading on water absorbency:

The effect of hectorite clay loading on the water absorbency of PS-g-PAA/HT superabsorbent composites is shown in Table 1 and Figure 2. It is observable that hectorite loading has an important effect on the water absorbency of the superabsorbent polymer composite. According to the results, the water absorbency increased as hectorite clay loading increased and decreased with further increasing of the hectorite clay loading. The water absorbency of the superabsorbent composite in distilled water and in 1.0 wt % NaCl solution increased from 33.83 g/g to 39.8 g/g and from 9.52 g/g to 15.2 g/g respectively, as 2% HT was introduced and when 6% HT was filling in the hydrogel matrix, water absorbency achieved the highest value which is 65.34 g/g in distilled water and 21.3 g/g in 1.0 wt % NaCl solution. This behaviour can be attributed by the presence of hydroxyl group (OH) on HT chain which can form hydrogen bond with water thus provides greater hydrophilicity effect and enhance the water absorbency (El, 2013). A further increase in hectorite loading to 8% leads to a decrease in water absorbency of 47.35 g/g in distilled water and 13.04 g/g in NaCl solution. This could be due the fact that hectorite act as physical filler which form physical and chemical crosslinker in the polymeric network (Ismail, 2012). Greater amount of hectorite result in the generation of more crosslinking point which increase the crosslink density of the superabsorbent polymer composite and decrease the elasticity of the polymer chains. The water absorbency decreased with decreasing polymer elasticity(Zhang, 2007).

4 Conclusion:

A superabsorbent polymer composite was prepared through emulsion polymerization technique based on partially neutralized acrylic acid (AA) grafted onto waste polystyrene foam using N,N'-Methylenebisacrylamide(NMBA), Ammonium Persulphate (APS) , and Span 60 as crosslinker, initiator and dispersant, respectively. From water absorbency analysis, the maximum water absorbency for PS-*g*-PAA/HT1 was 65.34 g/g in distilled water and 21.3 g/g in 1.0 wt % NaCl solution. The FTIR results indicate that the grafting polymerization took place among the polystyrene, acrylic acid and hectorite.

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