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Research Article
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Oil Sorbents Based on Methacrylic Acid - Grafted Polypropylene Fibers: Synthesis and Characterization

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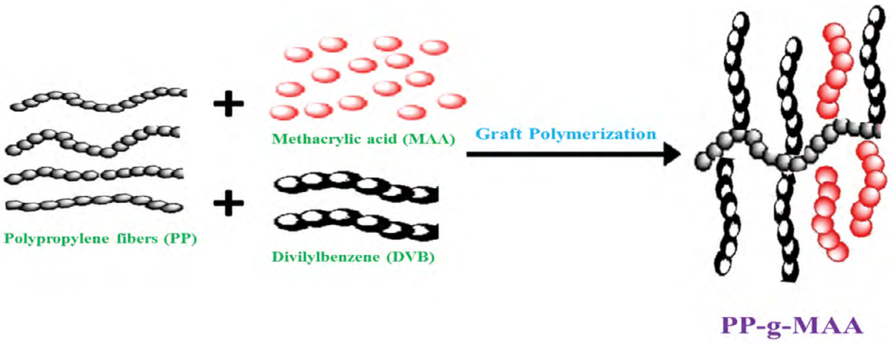
Abstract

PP-g-MAA oil sorption materials were synthesized by graft polymerization method of methacrylic acid (MAA) onto polypropylene fibers (PP) using divinylbenzene (DVB) as cross-linking agent. The virgin and grafted PP fibers were characterized by FT-IR, SEM, TGA, and DSC analysis. The results revealed that MAA was grafted onto PP fibers successfully. Factors affected the grafting yield (GY) were examined such as monomer concentration, initiator concentration, effects of temperature as well as reaction time. At the DVB and MAA concentrations of 1.5% and 2%, respectively, the maximum GY was obtained (73.1%). The obtained correlation coefficient (R^2) from well-known Langmuir sorption isotherm suggested well-fitting of the experimental data, which is assumed that at maximum coverage with identical sorption sites, there is monomolecular layer sorption on the PP-g-MAA surface. Oil sorption capacity of PP-g-MAA fibers was investigated which revealed a maximum oil sorption capacity (q_{max}) of 20.833 g/g. SEM pictures and FTIR results were taken for these materials under test.



Graphical Abstract

Keywords: Graft polymerization; PP fiber; PP-g-MAA; Oil sorption; Methacrylic acid; Divinylbenzene

Introduction

The world-wide energy resources can be separated into fossil fuel, nuclear fuel, non-renewable and renewable energy resources. Oil is still one of the most common and important fuel sources for transportation [1,2]. However, there is a great deal of leakage and spill risks during exploration, transportation, storage and use [3-6]. In the past twenty years, the impacts of oil spills and their effects had taken on much more importance and became a major form of pollution. Furthermore, oil spills can also affect environment, economy, tourism service as well as human health. These effects of oil spill depend on what and where type of oil was spilled (lake, river or sea). On the other hand, some typical large oil spills recently (Braer disaster in Scotland in 1993, Sea Empress oil spill in Wales in 1996 or Prestige accident in Spain 2002) has polluted thousands of coastline kilometers and inducing serious damages for the marine environment [7-9]. The reasons of spills are many, such as when a source of liquid petroleum hydrocarbon is released to the ecosystem, especially marine areas or land, by vehicle transport, vessel shipping or pipeline operation. Crude oil can be released from tankers, wells, offshore and shoreline waters. Hence, the rescue for oil spill issues, particularly marine oil spills where oil is released into the sea, ocean or coastal places, is extremely complicated, consuming a lot of manpower and material resources [10-12]. Using low cost oil sorbents is one of the most important approaches for recovering marine oil spills.

At present, numerous current techniques of oil removal, the use of oil sorbent is the most economical and effective method [13,14]. Some material has been widely studied, such as vegetable fibers (hay, straw) [15,16], polypropylene (PP) [17-19], kapok [20] as well as sugi bark [21]. In particular, their sorption capacities were compared with PP, that is, an ideal and commercial material for solving oil

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spill disaster on environment due to its hydrophobic and oleophilic properties. However, it still remains challenges to water-oil separation, eco-friendly ways of oil removing, free-floating oil skimmers as well as environmental concerns. In order to improve the oil sorption performance of virgin PP fiber, many modifications have been studied but graft polymerization is one the most effective methods to ameliorate its chemical properties and sorbent surface [22,23]. Some monomer such as butyl acrylate [19,24-26], lauryl methacrylate [27,28] have been successfully grafted.

To our knowledge, until now no existing literatures considered to conduct the graft polymerization onto PP fibers with methacrylic acid (MAA) using 2, 2'- azobisisobutyronitrile (AIBN) and divinyl benzene (DVB) as initiator and cross-linking agent, respectively. It is also reasonable to believe that the PP fiber hydrophobic-oleophilic characteristics could be attributed a waxy surface, led to an excellent oil absorbency and retention efficiency. Hence, in this research paper the sorption behavior of original PP and modified PP fibers had been examined. To determine the repeatability and consistency of the results, each experiment was repeated three times.

Materials and Methods

Materials

All the chemicals in this present article were of analytical grade (AR) and supplied from Sigma Aldrich, suitable for laboratory and general use. The original PP fibers were prepared by melt spinning method with some characteristics: diameter of 20-25 µm, tensile strength 350 MPa, specific gravity of 0.91 g/cm^3. They were cut into small parts (from 0.8 to 1.0 cm in length). All chemical reagents, such as methacrylic acid (MAA), 2, 2- azobisisobutyronitrile (AIBN), divinyl benzene (DVB), toluene, absolute ethanol and acetone were used as received.

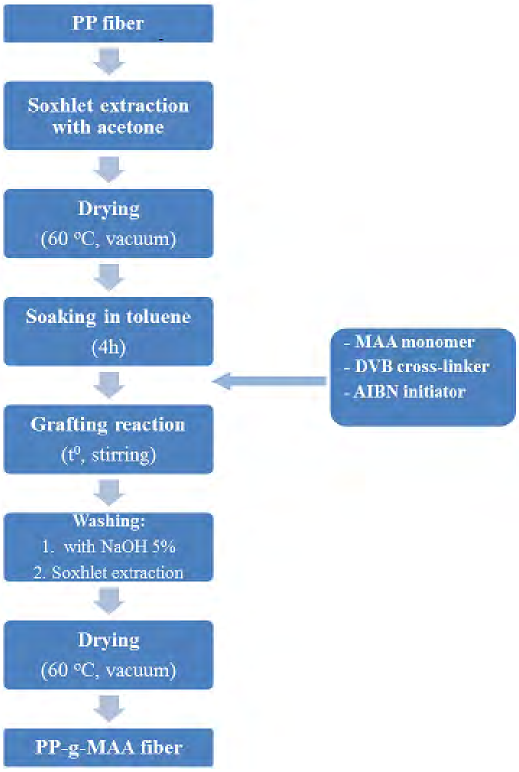
Synthesis of methacrylic acid-grafted PP fibers

The synthesis process was schematized in Scheme 1. Pure PP fiber was cleaned with acetone by chemically traditional Soxhlet extraction for 5 h, and then dried under vacuum at 60°C until a constant mass [29,30]. Grafting reaction was conducted in a 250 mL three-neck round bottom flask fitted with a reflux condenser, a magnetic stirrer and placed in a water bath. PP fiber (0.25 g) was soaked in 15 mL of toluene for 4 h and an exact amount of MAA monomer and DVB cross-linking agent was added. The system was purged with nitrogen gas during 10 minutes to remove oxygen, and then heated to a certain temperature under stirring. A calculated amount of AIBN was slowly added into the reaction mixture and stirred well. After 5 h, the MAA-grafted-PP fiber was taken out, washed with 5% NaOH solution which was prepared from a water/ethanol mixture (50: 50, v/v), then followed by a Soxhlet extraction with methanol for 24 h in order to remove all residual monomer and homopolymers. The product is and then dried under vacuum at 60°C until a constant mass. The graft yield (% GY) was determined by the following equation:

$$GY(\%) = \frac{W_g - W_0}{W_0} \times 100 \qquad (1)$$

Where: W_g and W_0 represent the weights of PP-g-MAA and initial PP fibers, respectively.

In order to investigate the influence of different parameters such as monomer concentration, initiator amount, cross-linker concentration, reaction temperature and time, we have considered one parameter as variable while the others as fixed. These independent variables were chosen in ranges that were generally used to be studied for grafting reaction.



Scheme 1: Process flow chart of polymer synthesis

Characterization

Fourier transform infrared spectroscopy (FTIR) spectra of virgin and grafted PP fibers were obtained on a Nicolet (IMPACT 410, USA) FTIR spectrometer in the range of 4000-400 cm^{-1}. The samples were ground with dried KBr powder and pressed into small discs prior to FTIR characterization. The spectra were collected by cumulating 32 scans at 4 cm^{-1} resolution. To study the surface morphology of the fibers, the images of scanning electron microscope (SEM) were conducted on JEOL 6390 (Japan) operating at an accelerated voltage of 10 kV. Sample surfaces were coated with a thin layer of gold. Thermal properties of the original PP and PP-g-MAA were evaluated by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) which were conducted on LABSYS DSC 131 and LABSYS TG/DSC 1600 (SETARAM), respectively. For DSC measurements, the samples were sealed in Aluminum crucibles of 30 µL by a sealing press and analyzed at a constant heating rate of 10°C/ min from ambient temperature up to 400°C under air atmosphere. TGA studies were carried out under air atmosphere with flow rate of 2.5 L/h. The samples were heated in a Pt crucible of 100 µL from ambient temperature up to 800°C at a uniform heating rate of 10°C/ min.

The maximum oil sorption capacity of experiments

For the sorption experiments, crude oil was added into a beaker containing sea water. Then, 2 g of PP-g-MAA fiber was placed onto the oil layer under shaking gently of 105 rpm to stimulate the motion of ocean waves. After certain time, the sorbent was taken out and drained for 2 minutes before weighing. The oil sorption capacity was calculated by using the equation following:

$$Q = \frac{(C_0 - C_1) \times V}{m} (g/g) \quad (2)$$

Where: C_0, C_1 are the initial and residual oil concentration after absorption, respectively (g.L^{-1}).

To determine the maximum sorption capacity of the sorbents, we investigated the oil sorption capacities with different oil concentrations: 15, 20, 25, 28.6, 30, 35, 40, 45, 50 and 55 mg/L, respectively. Sorption tests were carried out at 30°C for an equilibrium time of 30 minutes with a solution volume of 1 L.

Results and Discussions

Characterization of PP-g-MAA fibers

In Figure 1 represented the FTIR spectra of the virgin PP and the grafted PP fibers. Strong absorption bands at 2730-2980, 1450 and 1370 cm^{-1} in both spectra corresponded to the stretching and bending vibrations of C–H polypropylene [30,31]. Compared with the virgin PP, an obvious apparition of a new and intense absorption band at 1690 cm^{-1} was observed in the spectrum of grafted PP fiber due to the stretching vibrations of C=O, which indicated that the MAA group were successfully grafted onto the PP fibers by the grafting polymerization [24,32,33]. As illustrated in Figure 1, the FTIR spectrum of PP-g-MAA contained the characteristics bands for C – H in aromatic cycle of DVB at υ ~ 700-750 cm^{-1}. Moreover, we observed a shoulder peak at υ=1645 cm^{-1} attributed to C=C aromatic vibration band. These facts confirmed the presence of DVB cross-linker in the grafted product.

Another characterization by SEM images (Figure 2) also confirmed the grafting process. While the virgin PP fibers showed a quite smooth surface, the grafted PP fibers' surface became much rougher due to PMAA layer formed on the fiber surface [34,35]. In Figure 3 exhibited thermal analysis (TGA, DSC) results of virgin PP and grafted PP fibers, which indicated that the chemical modification of PP fibers caused changes in their thermal properties [2,36]. It can be seen from the TGA graphs (Figure 3a) that T_d 5% (the temperature at which the material decomposed 5%, weight loss=5%) increased strongly from 14°C for the initial PP to 275°C for the grafted PP fibers. This result shows that the grafting process enhanced clearly the thermal stability of the PP fibers [37,38]. This increase in thermal stability of PP fiber may be attributed to the abstraction of "labile" hydrogen from the tertiary carbon atom on the polypropylene backbone, followed by the grafting of PMAA chain at the site generated, which reinforced the structure as showed in Scheme 2.

Furthermore, the DSC thermogram (Figure 3b) of the grafted PP fibers revealed an exothermic peak at 227°C corresponding to the cross-linking process of the copolymer [39]. The melting peak can be seen in both diagrams at about 166°C but the fusion enthalpy of PP fibers has decreased lightly after grafting polymerization [40]. This result is due to the fact that the PMAA was incorporated into the polypropylene matrix as an amorphous component, which altered the melting behavior of the backbone polymer in the grafted sample.

Effect of temperature on the grafting degree

The reaction temperature was varied from 60 to 90°C while maintaining other reaction parameters in order to investigate the effect of temperature on the grafting degree. The results are shown in Figure 4, which indicates that the graft yield increased while increasing reaction temperature up to 80°C and then reduced if temperatures continue to rise. This can be explained by the fact that an increase in temperature induced an increase in the number of free radicals formed by the

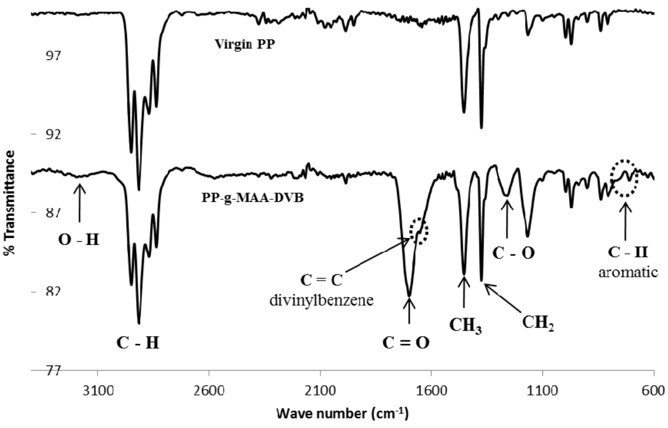


Figure 1: FTIR spectra of virgin PP and PP-g-MAA fibers.

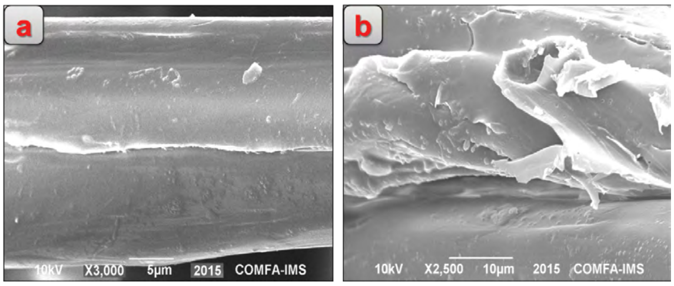
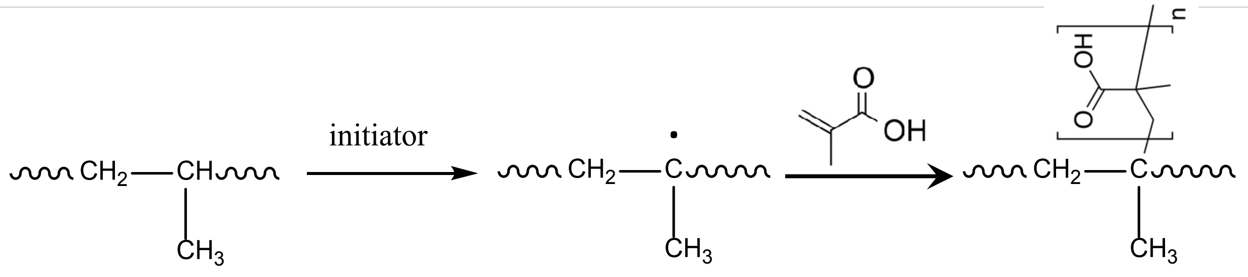


Figure 2: SEM images of PP (a), PP-g-MAA (b).



Scheme 2: The grafting process of PMAA onto PP backbone.

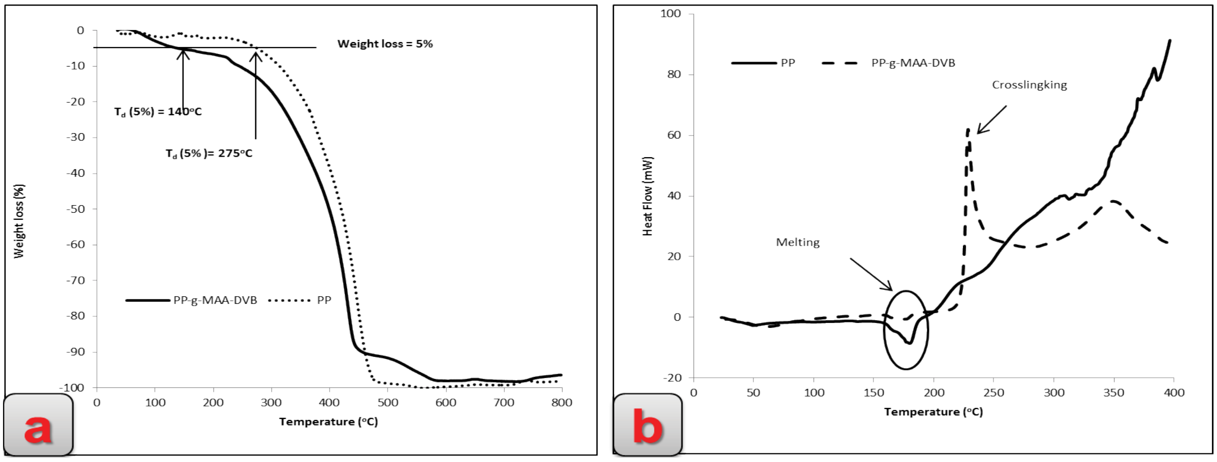


Figure 3: Thermal analysis of virgin PP and PP-g-MAA fiber: (a) TGA graphs, (b) DSC graphs.

thermo-decomposition of initiator, which results in more active sites on the PP backbone. Besides, high temperatures enhance the diffusion of the monomer to the active sites for polymerization [41]. However, when the temperature rose above 80°C, the initiator decomposed so fast that leads to an increasing formation of homopolymers which caused a decline in the expected amount of copolymer and consequently in the graft yield [42,43].

Influence of the reaction time

The grafting reaction was carried out for different time periods of 1, 2, 3, 4, 5, 6 h and the obtained results are shown in Figure 5 following. The reaction time has a remarkable effect on grafting yield as revealed Figure 5. With raising the reaction time, the monomer amounts grafted onto the fiber were increased. Graft copolymerization is relatively slow so that it needs time for the substances to react with each other. Therefore, having more time for reaction led to more attachment of monomers to PP fiber and more grafting yield. After exceeding an appropriate value of reaction time and consuming reactants, the grafting reaction tends to stop and reaches steady state. It can be clearly seen from the curve that the graft yield increased rapidly from 5% to 53% while the reaction time rose from 1 h to 5 h and the appropriate time to achieve higher grafting degrees in this study was determined as 5 h.

Effect of the monomer concentration

The reaction was carried out with a variation of MAA concentration from 1 to 10% and the result was shown in Figure 6. The curve indicates that the graft yield showed an upward trend to a maximum, then declines with the rise in monomer concentration [44]. At the monomer concentration of 2%, the GY was found to reach the highest value. At higher monomer concentration, higher viscosity of solution due to the homopolymers generated in reaction solution prevented the diffusion of monomer onto the fiber surface. Therefore, the grafting degrees decreased [45,46]. The optimum effect of monomer concentration is obtained with 2% v/v monomer concentration.

Effect of the initiator concentration

The effect of the concentration of the initiator AIBN on grafting level was studied by varying the AIBN concentration from 0.01 to 0,035 M. The results are shown in Figure 7. It can be observed that the grafting percentage increased rapidly up to a certain level and reached a maximum value of 73% at a concentration of 0.025 M of the initiator; beyond which it decreased lightly. The initial increase is maybe due to the availability of free radicals generated for grafting reaction. When the initiator concentration rises above a certain value, increased free radical concentration results in a homopolymerization which diminishes the graft copolymerization [23,47]. This result can be attributed to the fact that free radical generation process will increase with increasing levels of AIBN, which enhances the grafting degrees. Beyond the initiator concentration of 0.025 mol/L, a rise in the initiator concentration caused a decrease in grafting yield, which can be explained by the domination of homopolymerization process [48]. To sum up, the optimum condition for the grafting copolymerization of methylacrylic acid onto polypropylene fiber to achieve the highest grafting degree was given in the Table 1.

Effect of the cross-linking agent concentration on the oil sorption capacity

After determining the optimal conditions for the graft synthesis, DVB cross-linking agent was added to conduct a cross-linking process for the grafted copolymer [49]. Then, sorption experiments were conducted in an oil/sea-water system. The results are shown in Figure 8.

The Figure 8 presents the oil sorption capacity of PP-g-MAA obtained with different DVB concentrations. It turns out that in the beginning, the oil sorption capacity increased and reached a maximum level of 19.5 g/g with DVB concentration went up to 1.5%, but beyond this it fell down. This result can be explained as follows: a strong rising concentration of DVB causes a serious increased cross-linking level as well as a high density network, which reduces the space inside the gel, restricts the ability of the network to expand, therefore resulting in a decreased oil sorption capacity of the material [50,51].

Determination of the maximum absorption capacity

In this work, in order to present the equilibrium sorption behavior successfully a well-known sorption isotherm, namely Langmuir equations is tested to fit the equilibrium data. Furthermore, to determine the maximum oil sorption capacity of the grafted fibers PP-g-MAA, an isotherm Langmuir model was used and the calculated results were presented in Table 2 and Figures 9 and 10.

It was observed from Table 1 and Figures 9 and 10 that Langmuir sorption isotherm best describes the sorption of oil on the PP-g-MAA

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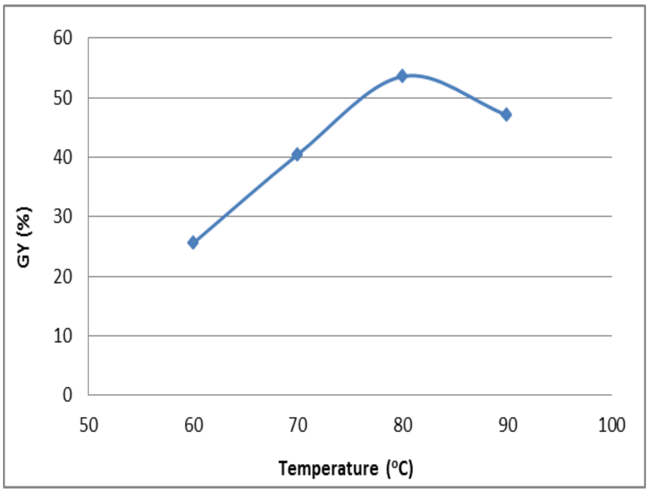

Figure 4: Effect of temperature on the GY: [MAA] = 2% (v/v), [AIBN] = 0.015 mol/L, reaction time 5 h

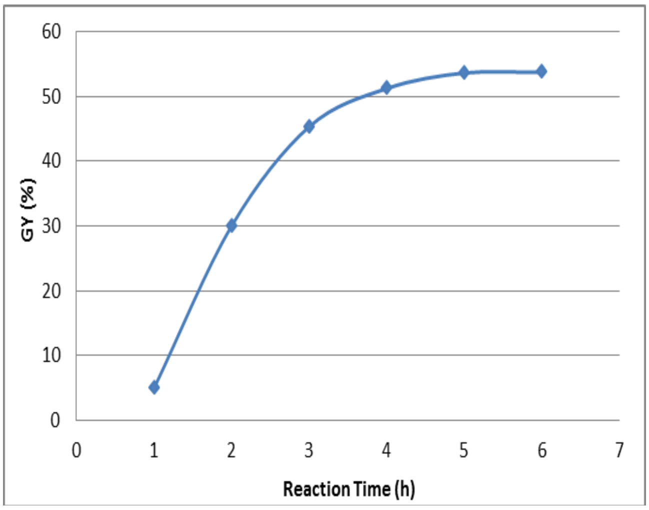

Figure 5: Influence of reaction time on the GY: [MAA] = 2% (v/v), [AIBN] = 0.015 mol/L, reaction temperature 80 °C

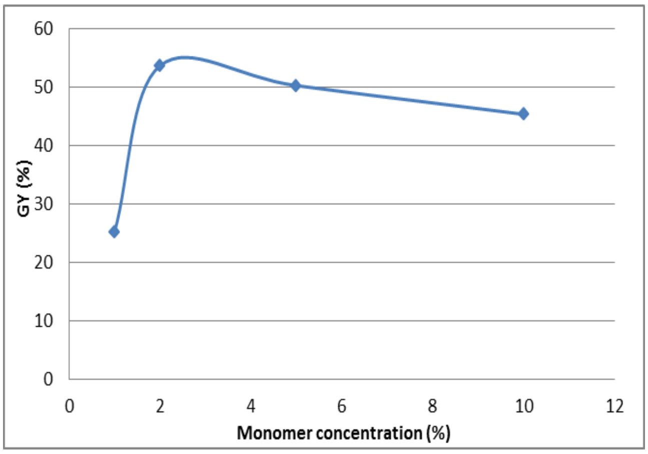

Figure 6: Effect of monomer concentration on the GY: [AIBN] = 0.015 mol/L, reaction temperature 80 °C, reaction time 5 h

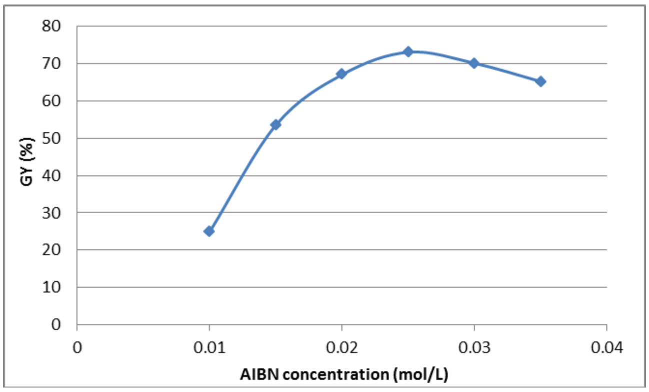

Figure 7: Effect of initiator concentration on the GY: [MAA] = 2% (v/v), reaction temperature 80 °C, reaction time 5 h

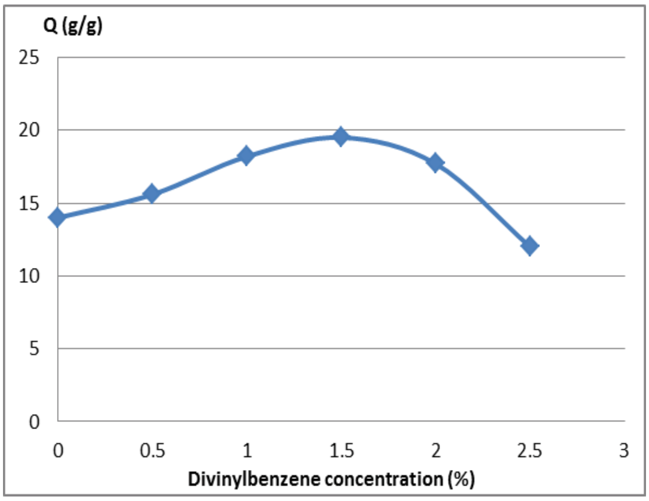

Figure 8: Influence of DVB concentration to the oil sorption capacity of PP-g-MAA.

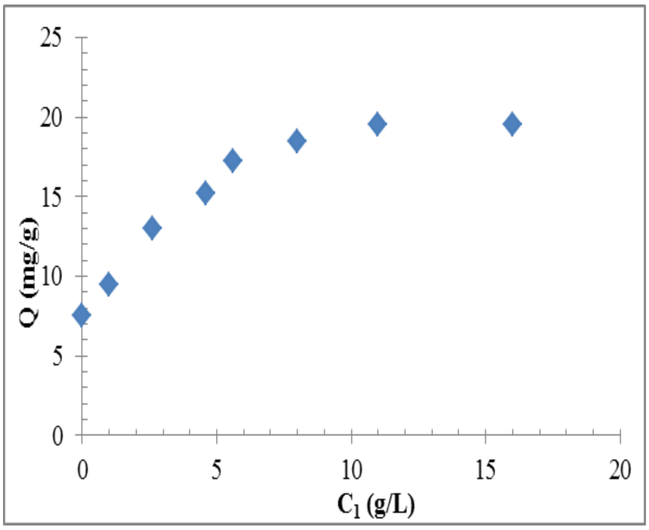

Figure 9: Absorption isotherm of PP-g-MAA.

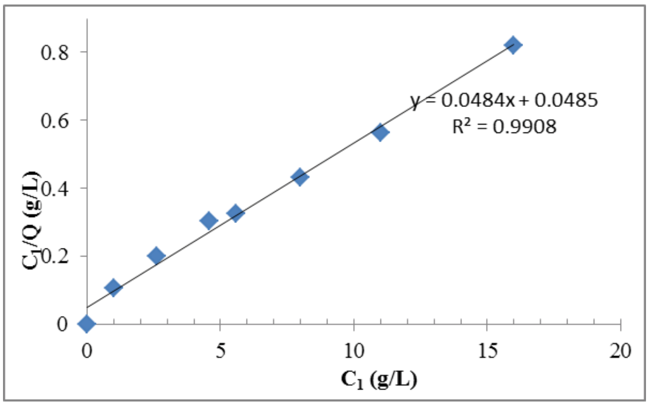

Figure 10: Linear graph for Langmuir constant determination.

	Temperature (°C)	Time (h)	MAA conc. (%, v/v)	AIBN conc. (mol/L)
Optimum value	80	5	2	0.025

Table 1: Main factors affecting the grafting degree.

Sample	C_0 (g/L)	C_1 (g/L)	Q (g/g)	C_1/Q (g/L)
1	15	0	7.5	0
2	20	1	9.5	0.105
3	28.6	2.6	13	0.2
4	35	4.6	15.2	0.303
5	40	5.6	17.2	0.326
6	45	8	18.5	0.432
7	50	11	19.5	0.564
8	55	16	19.5	0.821

Table 2: Calculated results following Langmuir model for PP-g-MAA.

fibers [52]. This can be demonstrated by a high linear correlation coefficient $R^2=0.9908$. This also proved that the sorption of oil on the grafted fibers was monolayer sorption [53,54]. From the obtained equation (Figure 10), we can identify the maximum oil sorption capacity is $\boldsymbol{Q}_{max}=1/0.048=20.833$ (g/g).

Conclusion

The key factors influencing the graft polymerization of methacrylic acid onto propylene fibers were investigated and we found optimal conditions to obtain the highest graft yield as follows: [MAA]=2%, [AIBN]=0.025 mol/L, temperature 80°C during 5 h. The presence of grafted MAA layer on the PP fibers was confirmed by FTIR measurements and SEM images. Furthermore, PP-g-MAA fibers performed interesting thermal properties such as an exothermic peak observed on its DSC diagram or a higher thermal stability compared with the one of virgin PP fiber. Besides, oil sorption studies following Langmuir sorption isotherm model gave good results with a high relevance between the experimental data and the model. The maximum oil sorption capacity of PP-g-MAA was 20,833 g/g after a sorption equilibrium time of 30 minutes.

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