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Synthesis and characterization of polypropylene glycol-graft-styrene

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ABSTRACT

Grafting polymerization based on polypropylene glycol (PPG) and styrene (St) was synthesized with different composition of styrene using the free radical technique in the presence of potassium persulphate as an initiator. The grafted copolymers (PPG-g-St) used different styrene composition (65/15, 65/25, 65/35), respectively. The grafted copolymers were investigated through FTIR (Fourier-transform spectroscopy), Differential scanning calorimeter and thermogravimetric analyzer. FTIR showed new peaks at 1450 cm^{-1} and 1135 cm^{-1} due to the grafting process of St on PPG. Thermal stability of grafted copolymer increases by increasing the ratio of styrene, while T_g decreases by increasing the ratio of styrene.

Keywords: Grafting, polymerization, styrene, copolymer, radical.

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Introduction

Polypropylene glycol (PPG) is a polymer with a broad industrial application, including biomedical mucoadhesive. In addition, PPG provides high chemical stability; and is a safe, biodegradable, and biocompatible polymer. Several researchers have distinguished chemical modification of polypropylene glycol via graft copolymerization. They have used different techniques, including anionic, cationic, free radical, and condensation polymerizations based on monomers type and direct attachment with functional polymer [1-7]. A series of polypropylene glycol-grafted-polyethyleneimines were synthesized with the grafting rate ranging from 9% to 19% [8]. Song et al., [9] reported that polypropylene glycol-grafted multi-walled polyurethane was synthesized based on the hydroxyl functionalized polyurethane through a two-step reaction. The obtained grafted copolymer can improve the rheological behavior of the polyurethane.

Ayman et al., [10] prepared PPG and grafted it with different molar ratios of maleic anhydride in the presence of dibenzoyl peroxide as a radical initiator. The obtained grafts were esterified different weights of polyethylene glycol monomethyl ether to produce nonionic surfactants. Do et al., [11] reported that the PPG-grafted polyimide precursor, poly (amic acid-*co*-amic ester), was synthesized via partial esterification of poly (amic acid) derived from pyromellitic dianhydride (PMDA) and 4,4′-oxydianiline (ODA) with bromo-terminated poly (propylene glycol) in the presence of K_2CO_3 in hexamethylphosphoramide and *N*-methyl pyrrolidone.

Maeda et al., [12] investigated the phase behaviour of graft copolymers in an aqueous solution. The graft copolymers consist of PPG side chains and N, N-dimethylacrylamide, N-vinylimidazole, and N-isopropyl acrylamide as backbones. Phase transition temperatures of copolymers increased with increasing the content of N, N-dimethyl acrylamide, and N-vinyl imidazole and with an increase in the degree of ionization of the incorporated N-vinylimidazole units. Murat et al., [13] prepared thermoplastic amphiphilic grafted copolymers based on PPG and different molecular weight of polyethylene glycol (PEG) in the presence of a base via a "grafting to" technique. The hydrophilicity of the amphiphilic copolymers increases with the increasing PEG content in the copolymer while the mechanical properties decrease.

The present work was designed to study the preparation and characterization of polypropylene glycol-graft-styrene via FTIR (Fourier-transform spectroscopy), thermogravimetric analyzer (TGA) and differential scanning calorimeter (DSC).

Experimental part

Materials: polypropylene glycol (PPG), styrene (St), potassium persulphate (KPS) and polyvinyl alcohol (PVA) were purchased from Sigma Aldrich.

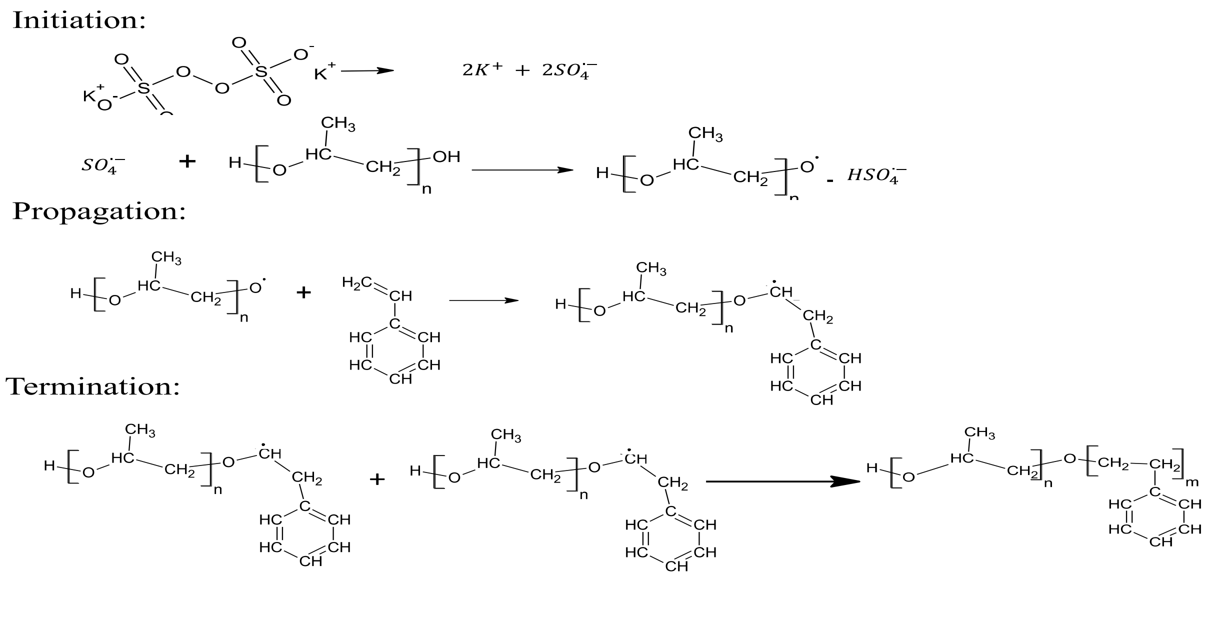
Preparation of PPG-Graft-St: Graft copolymerization was carried out in a 250 mL three-necked flask equipped with a thermometer, reflux condenser, and stirrer. The following procedure was used for the synthesis of grafting copolymers of polypropylene glycol (PPG) and styrene (St) with various ratios of St, i.e. [M1: (65: 15), M2: (65: 25), M3: (65: 35)]. PPG, PVA (0.1 gm) and water added to the flask and stirred continuously at a constant temperature of 60 °C. After the PPG was fully homogenous, the temperature of the system was strictly maintained at a required value. Freshly prepared potassium persulphate KPS solution about (5 mL) (0.74 mM) was added followed by dropwise addition of St. The reaction was conducted for two hours with stirring continued for another 20 min at room temperature.

Measurements: Infrared spectra were recorded on a Perkin Elmer 4000.0-400.0 cm^{-1} FTIR spectrophotometer. Thermogravimetric analyzer (TGA) was recorded on TGA/SDTA851^e, METTLER TOLEDO. Differential scanning calorimeter (DSC) were recorded on Pyris 1DSC, Perkin Elmer.

Results and discussion

Characterization of grafted copolymers

FTIR Spectra. FT-IR spectra of the PPG is shown in Figure 1a. Absorption peak at 3400 cm-1 due to the stretch OH group, peak at 3000 cm^{-1} for stretch C – H and 1100 cm^{-1} is ascribed to O – C group. Figure 1b presents the FTIR spectrum of grafted copolymer PPG-Graft-St.). The results showed new peaks at 1500 cm^{-1} and 2900 cm^{-1} correspond to (C = C) and (C-H) aromatic. It peaks at 1036 cm^{-1} which is attributed to stretch vibrations of C – O – C and confirm the point of grafting St unto PPG. Furthermore, the transmittance of the hydroxy group reduced more than 75% due to the grafting polymerization process as shown in Scheme 1.



Scheme 1 - Mechanism of grafted St on PPG

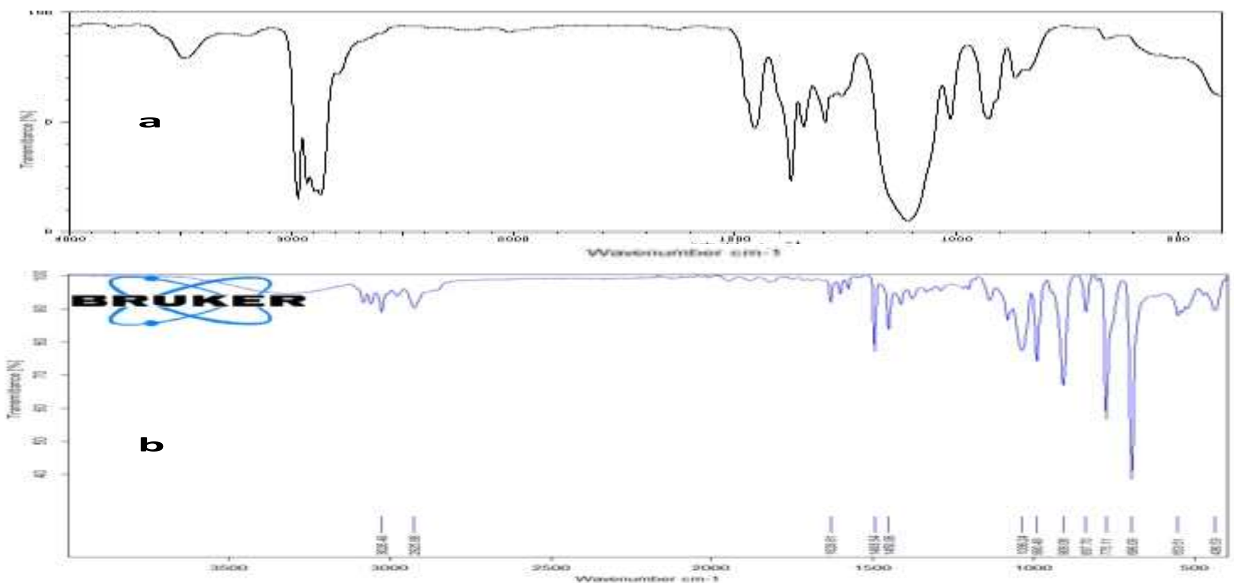


Figure 1 - FTIR spectra of (a) PPG and (b) PPG-Graft-St

TGA and DSC analysis

TGA in air at a heating rate of 10°C/min and DSC at about the same heating rate under nitrogen atmosphere is used to test the thermal activity of pure PPG and PPG-g-Styrene. Figure 2 – 5 show TGA and DSC analysis of pure PPG and PPG-g-Styrene. The thermal decomposition temperature of pure PPG and grafted PPG with a Styrene in different formulations is around 350 °C, as shown by TGA measurements.

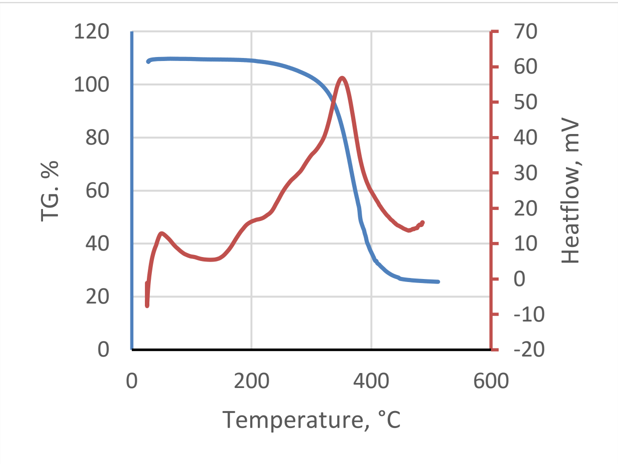


Figure 2 - TGA and DSC analysis of pure PPG

The TGA curves also show that pure PPG follows a one-step decomposition process, as shown in Figure 3. Between 200 and 500 °C occurs weight loss, which leads to total decomposition and CO_2 loss. Thermogravimetric curves can determine the quantitative phase composition of various substances. Therefore, our obtained samples are single-phase since they have one decomposition line. Thermal properties of samples shown in Table 1 illustrates the presence of styrene in the samples, with increasing of styrene in samples follows increasing in the temperature and amount of residual.

Table 1 - Thermal properties of samples obtained from TGA curve

Polymers	Temperature (°C)	Weight loss %	Residual %
PPG	200 - 420	74.32	25.68
M_1(65/15)	182 - 448	87.25	12.75
M_2(65/25)	235 - 467	83.54	16.46
M_3(65/35)	246 - 481	76.26	23.74

In DSC curves, peak area and the area bounded by the experimental curve and the baseline, which are proportional to the heat of reaction. Decomposition of prepared samples follow exothermic reaction. Moreover, a decrease of the peak area with increasing styrene indicates that the grafted polymer retains heat. The thermal characteristic from DSC analysis in Table 2, shows that the influence of styrene on glass transition, crystallization and melting temperatures is not big, but it still decreases [14, 15, 16, 17].

Table 2 - Thermal properties as samples from DSC curve

Polymer	T_g (°C)	Tc (°C)	Tm (°C)
PPG	58	144	354
M_1	58	112	354
M_2	54	114	360
M3	51	90	364

T_g: Glass translation temperature
T_c: Crystallization temperature
T_m: Melting temperature

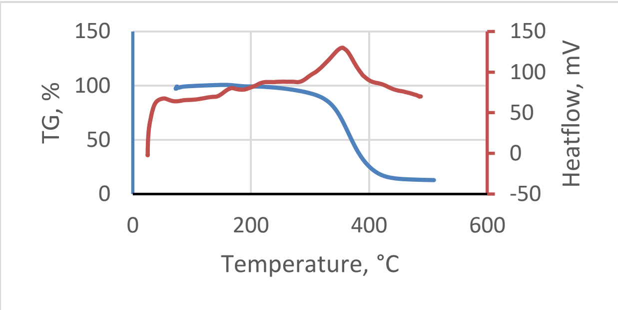


Figure 3. The TG and DSC analysis of PPG-graft-St (M1)

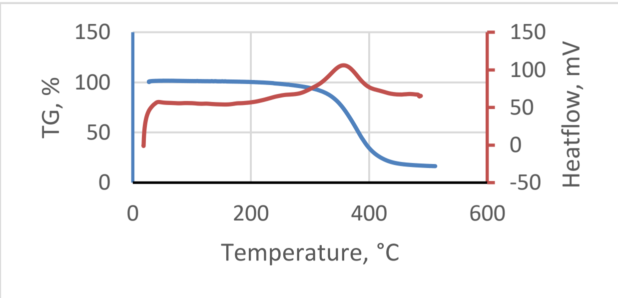


Figure 4. The TG and DSC analysis of PPG- graft -St (M2)

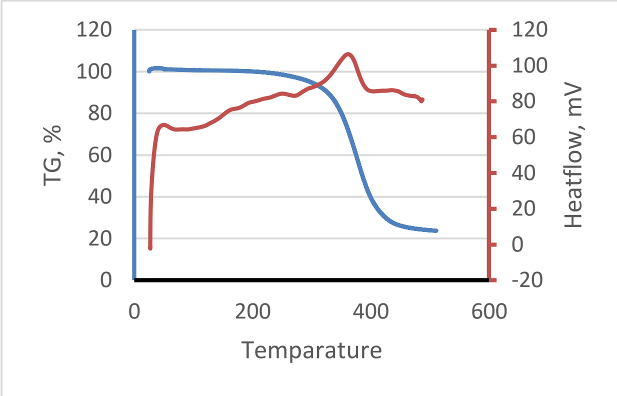


Figure 5. The TG and DSC analysis of PPG – graft- St (M3)

Conclusions

The grafted copolymers based on PPG and St were successfully prepared by grafting polymerization using free radical technique and potassium persulphate as an initiator. The grafted copolymer (PPG-Graft-St) showed an excellent thermal stability, however an increase in the ratio of styrene, thermal stability and T_g increase.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Стиролмен байланысқан полипропиленгликолдің синтезі және сипаттамасы

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ТҮЙІНДЕМЕ
Полипропиленгликоль (ППГ) және стирол (Ст) негізіндегі байланыстыру сополимеризациясы калий персульфаты инициаторының қатысуымен бос радикалды әдіспен әр түрлі құрамдағы стиролды қолдану арқылы синтезделді. Байланыстырылған сополимерлерде (ППГ-г-Ст) әр түрлі құрамдағы стирол (65/15, 65/25, 65/35) қолданылды. Байланыстырылған

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сополимерлер FTIR, DSC және TGA көмегімен зерттелді. FTIR (Фурье түрлендіру спектроскопиясы) 1450 см$^{-1}$ және 1135 см$^{-1}$ деңгейлерінде жаңа төбелерді көрсетті, бұл ППГ-нің СТ-ға байланысу процесін дәлелдейді. Байлнысқан сополимердің жылу тұрақтылығы (DSC, TGA) стирол пропорциясының өсуімен жоғарылайды, ал Tg сополимердегі стирол үлесінің артуына байланысты азаяды.

Түйін сөздер: Байланысқан сополимеризация, полимеризация, стирол, полипропиленгликоль, сополимер, радикал.

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АННОТАЦИЯ

Привитая сополимеризация на основе полипропиленгликоля (ППГ) и стирола (Ст) была синтезирована с использованием стирола разного состава путём свободнорадикальной методики в присутствии персульфата калия в качестве инициатора. В привитых сополимерах (ППГ-г-Ст) было использована стирол разного состава (65/15, 65/25, 65/35). Привитые сополимеры исследовали с помощью FTIR, DSC и TGA. FTIR (Фурье-спектроскопия) показал новые пики при 1450 см$^{-1}$ и 1135 см$^{-1}$, которые доказывают процесс прививки Ст на ППГ. Термическая стабильность (DSC, TGA) привитого сополимера увеличивается с возрастанием доли стирола, в то же время Tg уменьшается за счет увеличения доли стирола в сополимере.

Ключевые слова: Привитая сополимеризация, полимеризация, стирол, полипропиленгликоль, сополимер, радикал.

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