Basrah Journal of Science **Vol. 39(1), 135-148, 2021**

Synthesis of Branched Copolymers of Poly(1-hexene-*co*-maleic anhydride)

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Doi:10.29072/basjs.202119

Abstract

Poly(1-hexene-*co*-maleic anhydride) was synthesized by addition polymerization of 1:1 of 1-hexene and maleic anhydride using 1% benzoyl peroxide as initiator. The copolymer has been branched by different lengths of linear alkyl groups (C_{11}-C_{22}) via melt process esterification reaction with aliphatic alcohols using 1% para toluene sulfonic acid as catalyst. The prepared copolymers were purified and characterized by FTIR and NMR spectroscopy. All analyses indicate ring-opening reaction of anhydride group constituted backbone chains with aliphatic alcohols, which confirm the validity of the proposed chemical structures of copolymers. Molecular weight determination was confirmed by GPC and end group analysis (ASTM D1386-98) and found that 1-hexene monomer constitutes the largest portion in poly(1-hexene-*co*-maleic anhydride) backbone.

Article inf.

Received: 6/3/2021

Accepted

12/4/2021

Published

30/4/2021

Keywords

poly(1-hexene-*co*-maleic anhydride), addition polymerization, melt reaction, esterification reaction

1. Introduction

Maleic anhydride copolymers are polymers that contain maleic anhydride segment as essential component in copolymer backbone. They were widely studied and employed to manufacture a variety of products with potential uses in practically every field of industrial chemistry. They are used in production of unsaturated polyester resins [1], compatibilizer [2], plasticizer [3], epoxy curing and thickening agent [4], corrosion preventatives [5], detergent [6], dispersants [7], petroleum additives such as pour point depressant [8], pharmaceutics products as drug delivery [9]. The chemistry of maleic anhydride is very rich, reflecting its ready availability and bifunctional reactivity. The anhydride moiety can be hydrolyzed and esterified. Whereas, the potent double bonds can undergo cyclocondesation and copolymerization reactions, as long as monomer can't undergo radical homopolymerization [10]. However, the maleic anhydride copolymers are characterized by a great diversity in terms of physical and chemical properties and they can act either as a rigid or a flexible segment in a copolymer, depending on where the maleic anhydride unit in its open-ring or closed-ring form, and thus, they have been taken great attention by many researchers and also have a large library of derivatives with various synthetic techniques. Jincheng et al [11] used different molar ratios of maleic anhydride and 1-octadecanol to synthesis mono and dioctadecanoyl maleate via esterification reaction using para toluenesulfonic acid as catalyst. These products were then copolymerized with styrene and vinyl acetate monomers to produce random terpolymers, namely; poly(dioctadecanoyl maleate-*co*-styrene-co-vinyl acetate) and poly(octadecanoyl maleate-*co*-styrene-co-vinyl acetate). Crosslinking maleic anhydride copolymers by bifunctional-group polymer was established by Bettahar et al [12] who synthesized some mechanically stable hydrogels by thermally grafted poly(ethylene-*alt*-maleic anhydride) and poly(methyl vinyl ether-alt-maleic anhydride) with poly(ethylene glycol). Maleic anhydride copolymers are also found as anionic polyelectrolytes in poly(stilbene-*co*-maleic anhydride) and opening ring by ionic substance can impart charge densities to the copolymer [13].

A low polydispersity copolymer of ethylene, butylene, maleic anhydride and styrene was prepared using control copolymerization technique, reversible addition–fragmentation chain transfer. The RAFT agent based on poly(ethylene-*co*-butylene) used to synthesized block copolymers in which the second block is poly(styrene-*co*-maleic anhydride) [14]. Hemant and Dinakar [15] reported that copolymers of n-alkyl esters and maleic anhydride can be synthesized by two subsequent steps; esterification of n-alkyl alcohols (C_8-C_{22}) with oleic acid and methacryloyl chloride and then, copolymerization of resulting esters with maleic anhydride using (1% w/w) benzoyl peroxide as initiator. They also made further reaction of ring opening of anhydride constituted of these copolymers backbone using excess moles of n-alkyl alcohols. In the same manner, Mohammad et al [16] exposed hydroxyl groups on the surface of γ-alumina flaks to open the anhydride ring of some maleic anhydride and 1-alkenes copolymer. Others [17] used molar ratio of 1:2 of alkylated linoleic acid and maleic anhydride to produce tetra-esters copolymers. In this paper, we report on synthesis new series of maleic anhydride and 1-hexene copolymer derivatives having various alkyl branches lengths by addition polymerization and thermal solvent-free esterification reaction, copolymers structure analysis, molecular weight determination and monomers percentages.

2. General procedure

a. Materials

1-hexene supplied by Merck was purified by adding a dilute aqueous solution of sodium hydroxide (supplied by Fluka) to remove inhibitor, washed by distilled water, dried by anhydrous magnesium sulfate (supplied by BDH), and distilled at its boiling point. Maleic anhydride obtained from Sigma-Aldrich was recrystallized from dried chloroform three times. Benzoyl peroxide used as initiator also supplied by Sigma-Aldrich was recrystallized in acetone and then, precipitated by adding to the excess volume of water. P-toluene sulfonic acid (pTsOH), supplied by Sigma-Aldrich, was used as an esterification catalyst, which was dehydrated before use with toluene using Dean-Stark apparatus to azeotrope off the water at toluene boiling point. 1-undecanol obtained from Koch-Light Laboratories. Other aliphatic alcohols, 1-tereadecanol, 1-hexadecanol, 1-octadecanol, 1-eicosanol, and 1-docosanol were supplied by Sigma-Aldrich and used without purification. Toluene supplied by Fluka was dried by calcium chloride ($CaCl_2$) (Sigma-Aldrich), and was freshly distilled to obtain dry toluene before use. Tetrahydrofuran and n-hexane are supplied by BDH and RDH, respectively.

b. Instruments

FTIR spectra of all samples were recorded for the region between 400-4000 cm^{-1} by JSCO FTIR 4200, made in Japan, using KBr disk contains fine dispersion of the solid samples to be analyzed or thin-coating of liquid samples on KBr discs. NMR spectres were recorded on a Bruker Avance 400 spectrometer (400 MHz for ^{1}H-NMR, 100 MHz for ^{13}C-NMR) at 295 K using deuterated chloroform($CDCl_3$) or deuterated methyl sulfoxide-d6 (DMSO-d_6) as solvents. Chemical shifts (δ ppm) are reported in standard fashion with reference to either internal standard tetramethylsilane (TMS) (δH = 0.00 ppm), $CHCl_3$ (δH = 7.25 ppm) or DMSO (δH = 2.49 ppm). ^{13}C NMR spectra were recorded at RT in DMSO-d_6; chemical shifts (δ ppm) are reported relative to DMSO-d_6 (δC = 39.45 ppm) central line. While, at 77 ppm for $CDCl_3$. GPC with RI detector of

Waters Breeze™ 2 HPLC system and THF (as eluent) was used to the determined molecular weight and other related properties of the copolymer. The measurement was carried out using flowrate of 1 ml/min and sample volume of 100 µL at 25˚C.

c. Synthesis of poly(1-hexene-*co*-malic anhydride)

A mixture of 1 mole (15.24 g) of malic anhydride, 1 mole (13 g) of 1-hexene, and 1% w/w benzoyl peroxide in 250 ml of dried toluene was stirred mechanically in three-necks round bottom flask fitted with a condenser and glass tube as inlet gas section. The reaction mixture was degassed with argon for 10 mins at a moderated flow rate (~2-3 bubbles per second). Then, the reaction mixture was reflexed under argon for 6 hours. After that, the reaction was cooled down to room temperature, and the product of white solid powder was obtained, filleted, purified by dissolving in dried tetrahydrofuran and pouring in an excess volume of dried *n*-hexane, followed by filtration, washing with *n*-hexane three times and drying in vacuum at room temperature for 24 hours (yield: 89%)

2.4 Esterification of poly(1-hexene-*co*-malic anhydride)

1 mole (5 g) of poly(1-hexene-*co*-malic anhydride), 1.2 mole (8.875 g) of 1-undecanol, and 1% w/w pTsOH were charged into 250 three-neck flat bottom flask of the simple distillation apparatus. The reaction was taken place as a homogenous melt with stirring at 100°C under argon gas until distillation of water vapor is stopped. After that, distillation was carried out under a vacuum for one hour to extract residual water. The product was dissolved in tetrahydrofuran, reprecipitated in petroleum ether to remove unreacted alcohol, washed with distilled water to remove the catalyst, filtered, and dried in a desiccator under vacuum for 24 hours.Following the procedure above, longer aliphatic alcohols; 1-tetradecanol, 1-hexadecanol, 1-octadecanol, 1-eicosanol, and 1-docosanol, were also used for esterification of poly(1-hexene-*co*-malic anhydride). Table 1 listed the physical properties and yields of copolymers.

Table 1: List of poly(1-hexene-*co*-malic anhydride) derivatives used in the current study

Copolymer	Type of alcohol	Reaction time (h)	Physical behavior at 25°C	Yield (%)	Abbreviation
Poly(1-hexene-*co*-diundecyl maleate)	1-undecanol	4	Clear yellow oil	95.2	B1
Poly(1-hexene-*co*-	1-tetradecanol	4	Clear yellow oil	92.9	B2

ditetradecyl maleate)					
Poly(1-hexene-*co*-dihexadecyl maleate)	1-hexadecanol	4	White to yellow soft wax	94.8	B3
Poly(1-hexene-*co*-dioctadecyl maleate)	1-octadecanol	4	White to yellow hard wax	92	B4
Poly(1-hexene-*co*-diicoscyl maleate)	1-eicosanol	5	White to yellow Solid	92.1	B5
Poly(1-hexene-*co*-didocoscyl maleate)	1-docosanol	5	White to yellow Solid	93.5	B6

2.5 Group acid analysis method

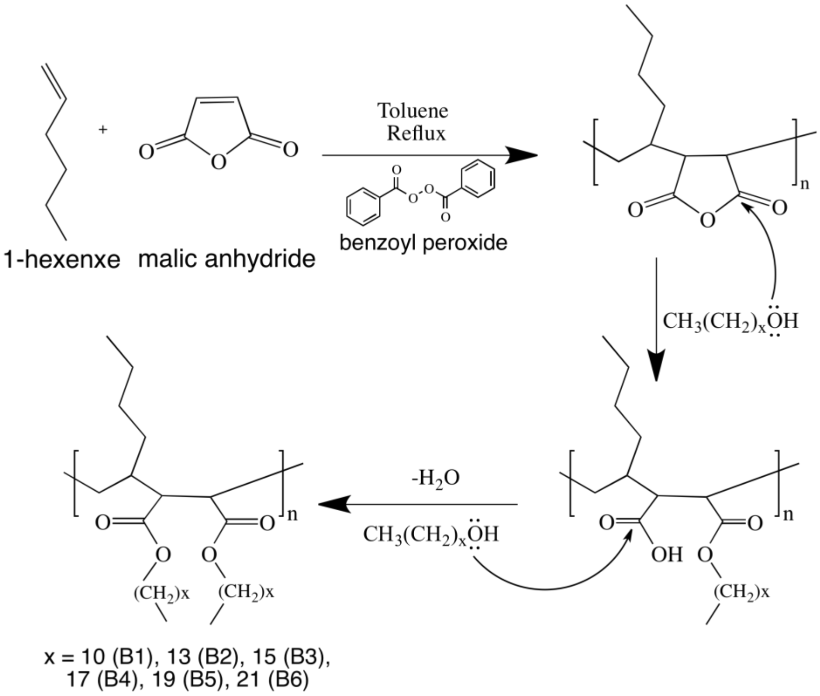
1 g of polymer and 40 ml of xylene were added into 250 ml conical flask and heated by hot plate until all polymer dissolved entirely. 3 to 5 drops of phenolphthalein indicator solution were added and then, the hot solution was titrated with 0.1 N of ethanoic potassium hydroxide (dissolving 6.6 g of potassium hydroxide in 5.6 g of distilled water, dilution to 1000 ml by 95% ethanol and standardization with 0.1 N HCl). The end point was determined when the pink color remains, at least, 10 sec. The molecular weight of polymer was calculated by amount of milliliters of potassium hydroxide solution required to neutralize 1 g of polymer using mathematical terms [18]:

$$M.wt_s = \frac{W_s * 1000}{C_{KOH} * V_{KOH}} * n \qquad (1)$$

Where $M.wt_s$ is the average molecular weight of the polymer (g/mol), W_s is polymer weight (g), n is KOH moles number required to neutralized acidic groups in one polymeric chain, C_{KOH} is KOH concentration (mol/L) and V_{KOH} is volume of KOH solution (ml) required to neutralized specific amount of polymer.

3. Results and discussion

Scheme 1 shows a schematic diagram of copolymers synthesis in the present study. Reactions proceed in three subsequent steps. First step involves synthesis of copolymers containing five-member anhydride ring. Second step is esterification reaction of alkyl alcohols with copolymer anhydride group and the third step is esterification reaction of alkyl alcohols with remaining acid groups using pTsOH.



Scheme 1. Schematic diagram of the synthesis of poly(1-hexene-co-maleic anhydride) derivatives

3.1 FTIR analysis

Figure 1 shows the FTIR spectrum of poly(1-hexene-*co*-maleic anhydride) and its corresponding monomers. Maleic anhydride is a heterocyclic compound that contains a double bond (C=C) between the second and third carbon shows band at 1643 cm^{-1}. Such double bond leads to increase stretching of (=C-H) to 3127 cm^{-1}. Two strong bands emerged at 1781 cm^{-1} and 1856 cm^{-1} are assigned to symmetric and asymmetric stretching of carbonyl (C=O) or anhydride group, respectively. 1-hexene shows four bands; at 3100 cm^{-1} assigned to (=C-H) bond stretching, at 2950 cm^{-1} and 2873 cm^{-1} related to methyl and methylene group, respectively, and at 1643 cm^{-1} assigned to (C=C). However, FTIR of poly(1-hexene-*co*-maleic anhydride) indicated the continued existence of two strong bands, at 1781 cm^{-1} and 1856 cm^{-1}, of carbonyl (C=O) or anhydride group, in addition to bands between 2950 cm-1 and 2850 cm^{-1} of methyl and methylene groups [19-21]. But, it also indicated that significant reduction in absorption of (C=C) double bond at 1643 cm^{-1} and a noticeable absence of (=C-H) bond stretching existed in both reactant monomers, which confirm the polymerization was successfully achieved by free radical polymerization while keeping anhydride group intact. Figure 2 illustrated the FTIR spectrum of the esterified products (B1-B6), which indicates the appearance of new strong bands at 1731 cm^{-1} related to carbonyl (C=O) group stretching of ester group and the two bands appeared at 1168 cm^{-1} and 1380 cm^{-1} assigned to (C-O) bond stretching of the ester group. It also indicated the absence of anhydride absorption peaks, at 1781 cm^{-1} and 1856 cm^{-1}, and that confirmed esterification of acid anhydride groups containing poly(1-hexene-*co*-maleic anhydride) was achieved by alkyl alcohols. Pattern spectra of all esterified copolymers are comparable with a considerable decrease in ester/methylene groups when the reaction was conducted with longer aliphatic alcohols.

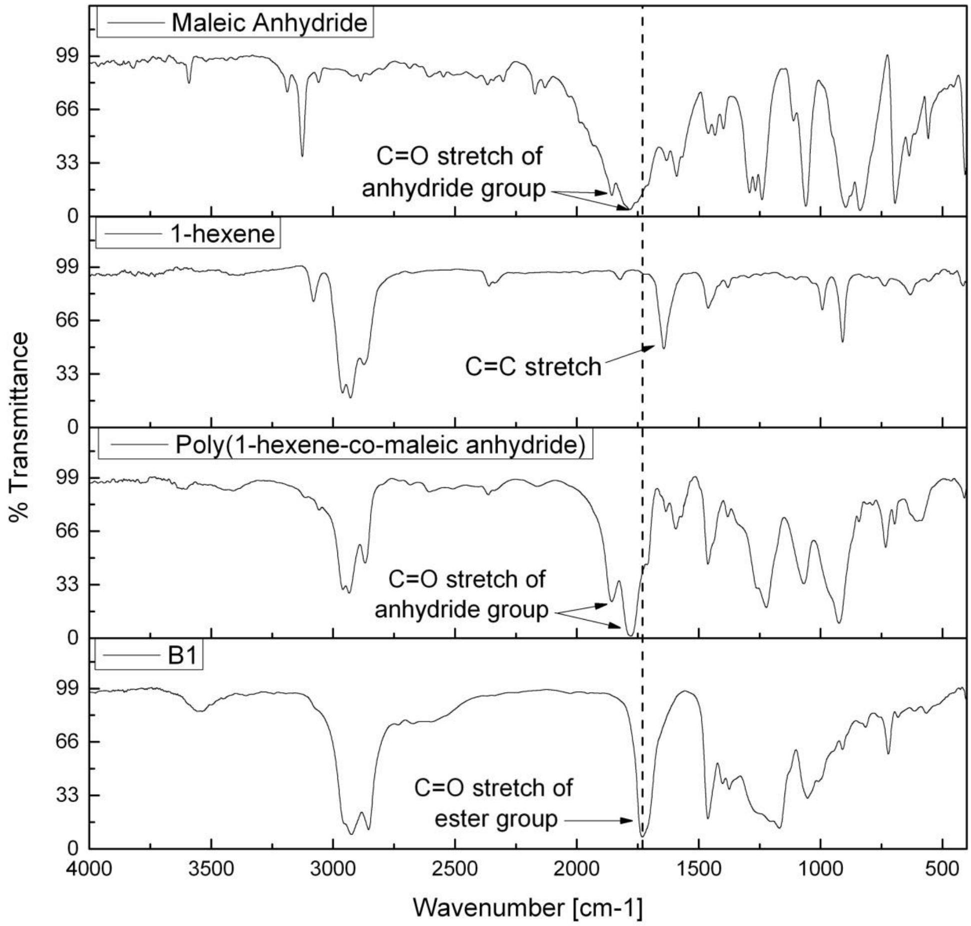


Figure 1. FTIR spectra of poly(1-hexene-*co*-maleic anhydride) and its corresponding monomers.

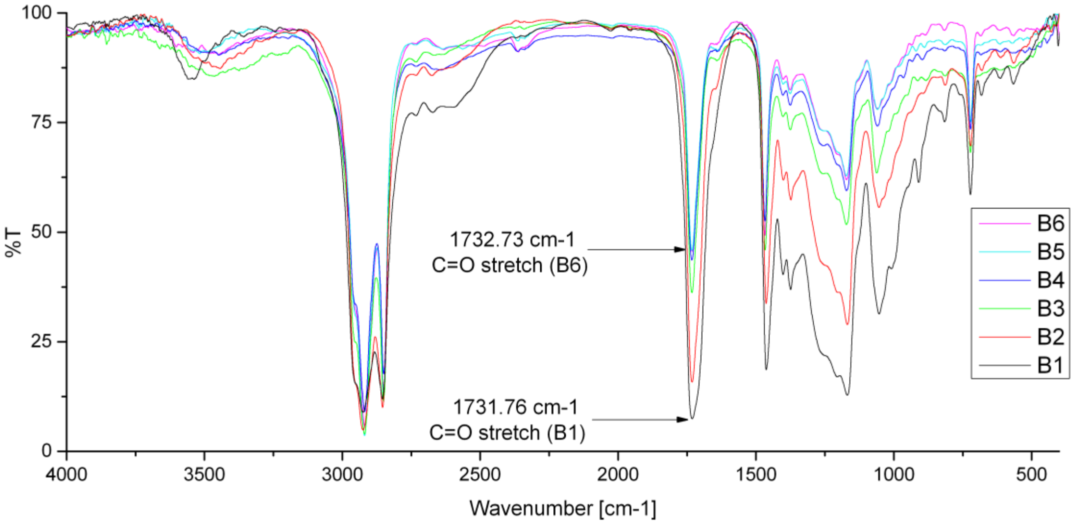


Figure 2: FTIR spectra of poly(1-hexene-*co*-maleic anhydride) derivatives showed the decrease in the intensity bands of the carbonyl group

3.2 ^{1}H NMR analysis

^{1}H NMR spectrum of poly(1-hexene-*co*-maleic anhydride) is shown in Figure 3. It indicated the appearance of the four groups; methyl group (a) at the end of branches which appears as a signal at 0.8 ppm, aliphatic methylene group (b) neighboring methyl group which has a signal at 1.3 ppm, group (c) protons which constituted copolymer backbone, exhibits signal peaked at 1.8 ppm, and finally, group (d) protons although it constituted copolymer backbone, directly attached with carbonyl group which that leads to appear as a broad peak at the higher chemical shift, between 2.9 and 3.5 ppm [22, 23]. Many researchers reported that polymerization of maleic anhydride ring results in peak broadening [24-26]. Other weak signals between 7.1 ppm and 7.3 ppm related to protons of the aromatic ring of benzoyl groups because of benzoyl peroxide decomposition.

The ^{1}H NMR spectrum of all esterified copolymers (B1-B6) have almost the same pattern spectrums. The signal at 0.9 ppm represents the protons of a methyl group (a). While the signal at 1.3 ppm assigned to protons of a methylene group, (b) and its density increased significantly with the increasing length of the pendant alkyl group. The analysis also indicates the signal of backbone protons (c) at 1.7 ppm. Ring-opening reaction by esterification reaction of anhydride group was characterized by three indications; the first one is a proton (d) which shifting to 2.8 ppm. Authors [25, 27] pointed that the opening ring of the anhydride segment results in peak shifting of hydrogen (d) towards higher magnetic fields. The other ones involve appearing new signals at 4.1 ppm and 3.7 ppm related to the proton of a methylene group (f) and (e), respectively.

3.3 ^{13}C NMR analysis

Figure 4 shows the ^{13}C NMR spectrum of poly (1-hexene-*co*-maleic anhydride). The signal at 167 ppm was attributed to carbonyl carbon (C_6). Many researchers indicated the appearance of carbon (C_5), which is part of the copolymer backbone and also part of the five-membered ring, at a chemical shift of 40 ppm [28, 29] However, such a chemical shift also contains a strong peak of deuterated DMSO-d_6. The existence of methyl carbon (C_1) of the branch was also observed as the signal at 14 ppm. The signals, at 23 ppm represents adjacent methylene carbon (C_2), at 32 ppm assigned to methylene carbon (C_3), and at 28 ppm related to carbon (C_4), and weak signals in the range of 123 to 130 ppm assigned to methylene carbon of benzoate ring at the ends of polymer chains.As mentioned in literature [30], esterification of anhydride ring containing copolymer can characterize by the appearance of the signal between 61 ppm and 65 ppm, and thus, as shown in Figure 4, the appearance of a peak at 65 ppm in B1 is a good indication of achieving esterification reaction in which methylene carbon (C_9) becomes adjacent of oxygen of ester group. Also, indicating methylene carbons (C_3 and C_8) at the lower chemical shift, between 31 and 33 ppm. Other signals involve peaks between 28 and 30 ppm assigned to carbon (C_4), peak at 165 ppm related to carbonyl carbon (C_6), peaks at 39, 25, 23, and 14 ppm assigned to carbon (C_5), (C_7), (C_2) and (C_1), respectively. Analysis of copolymer esterification was also extended to other copolymer B2-B6, and found that esterification with longer linear alcohols leads to a significant increase in peak amplitude of carbon (C_4).

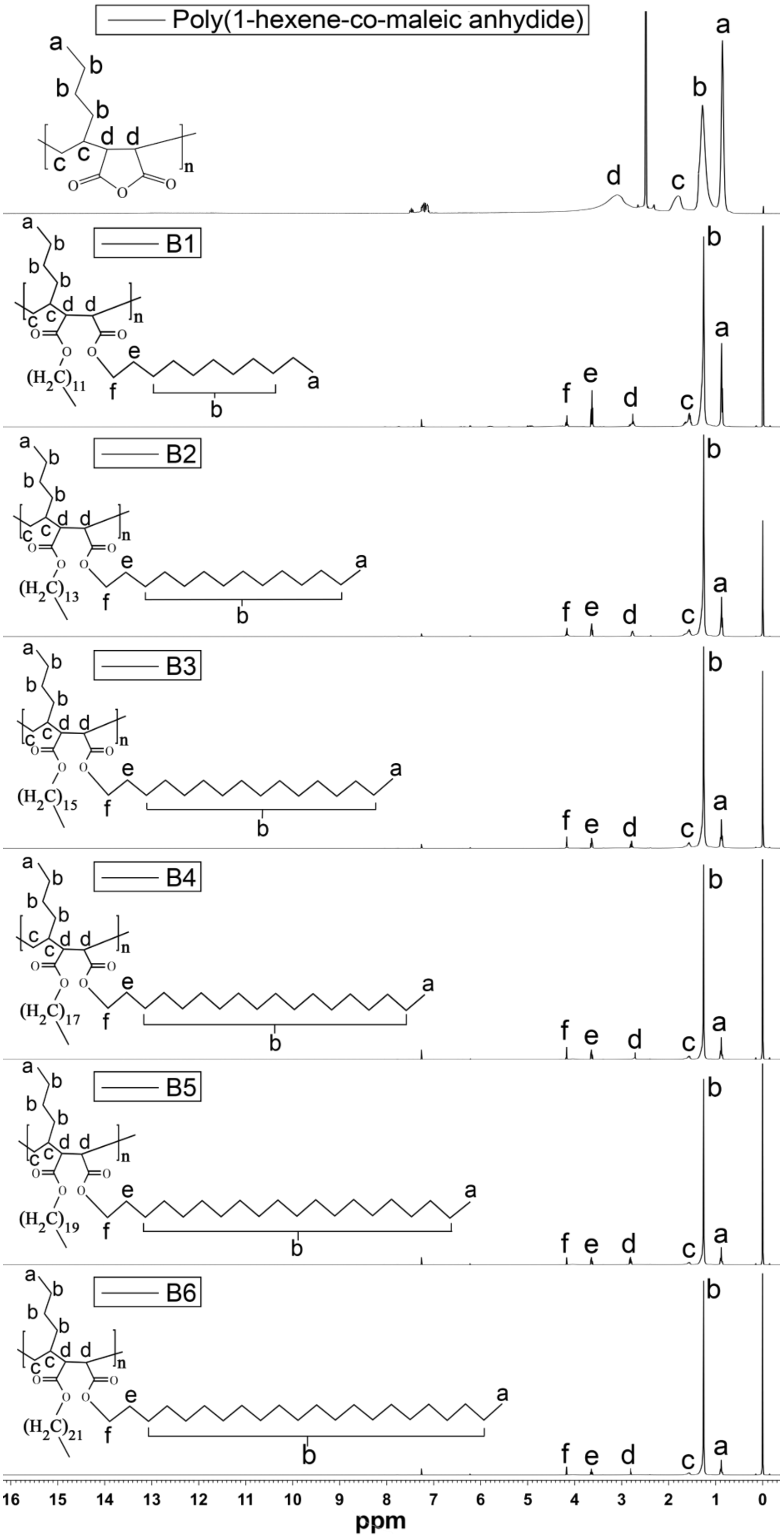


Figure 3: ^{1}H NMR of poly(1-hexene-*co*-maleic anhydride) and its copolymer derivatives (B1-B6).

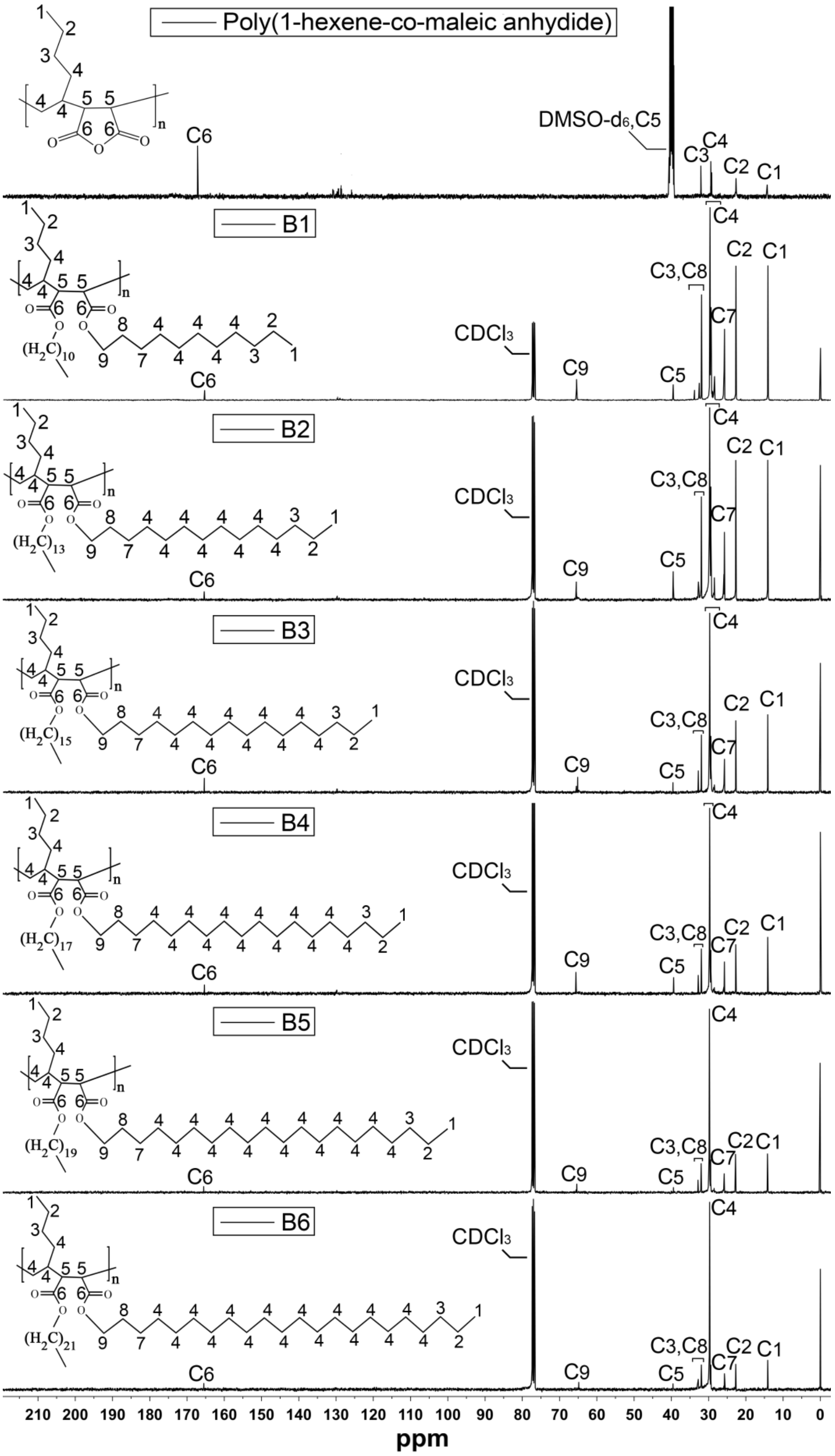


Figure 4: ^{13}C NMR of poly(1-hexene-*co*-maleic anhydride) and its copolymer derivatives (B1-B6).

3.4 GPC and group acid analysis

The molecular weight determination via GPC analysis of the unesterified copolymer is shown in Figure 5. It was found that PDI was 1.70. and, by the aid of acid group analysis (ASTM D1386 – 98), the copolymer contains 39.18% maleic anhydride and 60.82% 1-hexene.

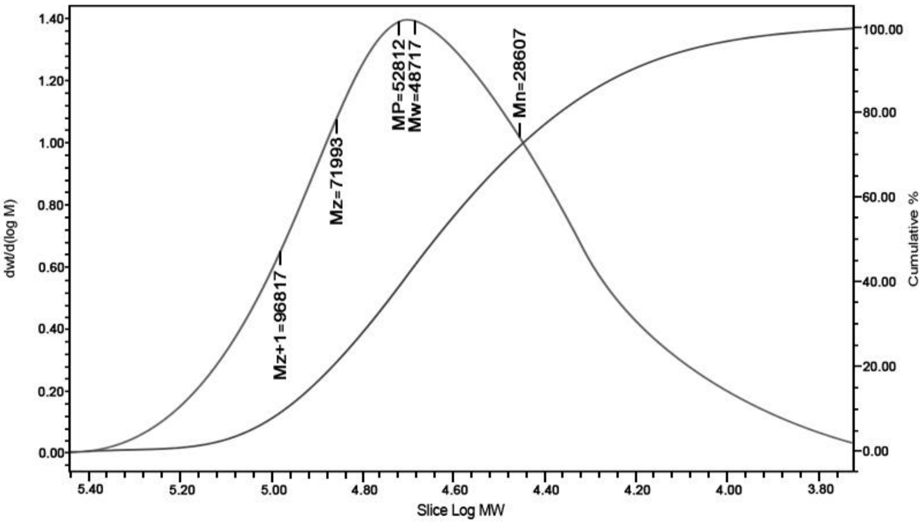


Figure 5: GPC analysis of poly(1-hexene-*co*-maleic anhydride)

4. Conclusions

In this study, copolymers of poly(1-hexene-co-maleic anhydride) containing different branch lengths were successfully synthesized and characterized by FTIR, ^{1}H NMR, ^{13}C NMR, end group acid and GPC analysis. The analysis showed that the resulting poly(1-hexene-co-maleic anhydride) is in a good range of polydispersity and contains about 60.82% of 1-hexene and 39.18% of maleic anhydride. They were also showed that attaching side alkyl groups to copolymer main chain was thermally accomplished as a melt via esterification of anhydride acid group constituted copolymer backbone with linear aliphatic alcohols, in which this nucleophilic attack of alkyl alcohols to anhydride acid group is mainly influenced by reaction time and molecular weight of alcohols.

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المستخلص

تم تحصير البوليمر المشترك من 1-هكسين والحامض الماليك اللامائي عن طريق بلمرة الأضافة ١:١ من 1-هكسين وحامض الماليك اللامائي باستخدام ١٪ بيروكسيد البنزويل كبادئ لعمليه البلمرة . تم ربط البوليمر المشترك بأطوال مختلفة من مجموعات الألكيل الخطيه $(C_{11}-C_{22})$ بواسطه تفاعل الأسترة كمنصهرات مع الكحولات الأليفاتية باستخدام ١٪ بارا التولوين حامض السلفونيك كمحفز ، تمت تنقية البوليمرات المشتركة المحضرة وشخصت بواسطه مطيافيه الأشعة تحت الحمراء FT-IR والرنين النووي المغناطيسي NMR ، وتشير جميع التحاليل إلى اتمام تفاعل فتح الحلقة لمجموعة الحامض اللامائي المكونة لسلاسل العمود الفقري للبوليمر المشترك مع الكحولات الأليفاتية ، والتي تؤكد صحة التراكيب الكيميائية المقترحة لهذه البوليمرات المشتركة. وتم أيضا تأكيد تحديد الوزن الجزيئي من بواسطة كروماتوغرافيا الاستبعاد بالحجم الهلامي GPC والمجموعة النهائية (ASTM D1386-9) ووجد أن المونيمر 1-هكسين يشكل الجزء الأكبر في العمود الفقري من هذا البوليمر المشترك