COMPARATIVE ANALYTICAL STUDY OF POLYISOBUTYLENE SUCCINIC ANHYDRIDE-MODIFIED TRIETHANOLAMINE EMULSIFIER

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ABSTRACT

An analytical comparative study was performed between the prepared emulsifiers polyisobutylene succinic anhydride (PIBSA) and the commercial LZ2735 and LZ2745 materials. The PIBSA-1, -2 and PIBSA/TEA were prepared by reacting the reactive polyisobutene (PIB) with maleic anhydride via isothermal heating at 250°C for 10 h. Then, PIBSA was heated with triethanolamine at 80°C for 2 h to give PIBSA/TEA. The structures of the prepared PIBSA-1, PIBSA-2, PIBSA/TEA, PIBSA/TEA-1, PIBSA/TEA-2, LZ2735 and LZ2745 were characterized by FTIR, ^{1}H-, ^{13}C-NMR spectroscopy and Gel permeation chromatography (GPC). The FTIR spectra of PIBSA-1 and -2 showed absorption carbonyl bands at 1711 and 1783 cm^{-1} due to carbonyl anhydride stretching vibrations of the succinic anhydride (SA), whereas a relevant band appeared at 1732 cm^{-1} for LZ2735 and LZ2745. PIBSA/TEA was identified by a hump of amine group around 3351 cm^{-1}. ^{13}C-NMR of the PIBSA-1 and -2 indicated signals at δ 57.6 and 59.5 ppm. The GPC showed polydispersity index which used as a measure of the broadness of a molecular weight distribution of our samples together with the presence of a significant amount of number average molecular weight (M_n). The amber colored products were viscous and the prepared emulsions were used for blending the emulsion explosives, detergents, cosmetics, paintings to remove lead from polluted waste water.

Keywords: PIBSA. NMR. FTIR. GPC. MP-AES.

1. INTRODUCTION

Lubrizol Company introduced the PIBSA-based emulsifiers as explosives in the 1980s. Since the early 1990s, PIBSA-based surfactants have been used to afford kinetic stability to high internal phase (HIP) of water-in-oil emulsions [1–4]. The chemical structures of these amphiphilic molecules determine the nature of their interaction with the oil–water interface, the ability to form reverse micelles in the oil phase and their time-stabilizing efficiency [5, 6]. The effective concentrations of these surfactants are usually determined by the emulsion stability limits, and typically are well in excess of their critical micelle concentration (CMC), to afford total surfactant concentration in the oil of 0.01–1.5 wt% [6, 7]. Whilst the active surfactant loading at the water–oil interface is almost independent of dilution, the concentration of reverse micelles in the oil phase is excess concentration dependent and proved to be critical for the HIP

emulsion stability, especially at elevated temperatures [8,9]. The industrial method of production of PIBSA-based surfactants is typically a two-step process, starting from the PIB followed by the addition of maleic anhydride (MAH) with further substitution/addition of other functional groups. Further thermal treatment of the open ring structure can result in cyclization to the PIBSA/TEA surfactant. The second method included chlorine-free radical process .The viscosity of the oil phase is one of the key physicochemical parameters and can be greatly affected by the structure of the surfactant polar head group, average mass number (M_n), average molecular weight (M_w), the degree of functionalization and content of surface inactive products [10].

In this work, PIB functionalized with maleic anhydride was carried out at a temperature of 250°C for 10 h, then the product (PIBSA) could react with TEA in presence and

absence of paraffin oil. The synthesized materials were analyzed by FTIR,^{1}H NMR and ^{13}C NMR spectroscopy to probe the interaction between PIBSA and TEA.

2. EXPERIMENTAL

2.1. Raw materials and methods

The highly reactive poly isobutylene (M_n = 2400 g/L) was purchased from Shanghai Minglan Chemical Company. Maleic anhydride (M_w = 98.08 g/L) and Triethanolamine (TEA) (M_w = 150 g/l) were supplied by LOPA Company. Paraffin oil was provided by Misr Petroleum Company. Lead Acetate trihydrate (M_w = 379.33g/L) was purchased from Biotech for Laboratory Chemical.

2.2.1 Preparation of PIBSA-1 and PIBSA-2

4.8 g (0.002 mole) of highly reactive polyisobutylene (M_n = 2400) were mixed with 442.47 g (0.0045 mole) of maleic anhydride in a special vessel with continuous stirring using magnetic stirrer (PIBSA-1). Also, 6 g (0.0025 mole) of poly isobutylene were mixed with 1000 mg (0.010 mole) of maleic anhydride in a closed vessel under magnetic stirring (PIBSA-2). Both vessels were equipped with Liebig condenser and purged with nitrogen gas for 30 min. The reaction slurries were continuously heated on a sand bath at 250 °C for 10 h. The obtained products were colored honey.

2.2.2   Preparation of PIBSA/TEA, PIBSA/TEA-1 and -2

1000 mg (0.0004 mole) of PIBSA were added to 735 mg of paraffin oil and 145 mg (0.00097 mole) of triethanolamine in vessel (1) under continuous stirring. Meanwhile, 1000 mg (0.0004 mole) of PIBSA were added to 730 mg of paraffin oil and 135 mg (0.00090) of triethanolamine in vessel (2) under vigorous magnetic stirring and purging nitrogen atmosphere. Vessels (1) and (2) were heated in sand bath already set at 80 °C for 2 h to get PIBSA/TEA-1and PIBSA/TEA-2, respectively. The latter preparation was carried out again but without adding paraffin oil (PIBSA/TEA) for comparative purposes. Thus, 2498 mg (1 mole) of PIBSA were added to 150 mg (1 mole) of triethanolamine TEA under continuous stirring at 120 °C for 2 h to yield PIBSA/TEA.

System of reaction

2.3. Analytical Instrumental

FTIR spectra were measured using a Bruker Spectrometer, Model M-IR, Germany.

^{1}H and ^{13}C-NMR spectra were recorded on a Bruker 400 MHz instrument. All chemical shifts in the text, tables and figures were reported in δ (ppm), referred to tetramethylsilane (TMS) as external standard and referenced to the residual proton and carbon signals DMSO as solvent which appear at δ_H 2.50 ppm and δ_C 39.51 ppm, respectively. Routine analysis generally involved in 1D carbon, NMR spectrometer (Varian Mercury console).

Gel permeation chromatography (GPC) was carried out using a Hewlett–Packard 1100 series chromatograph interfaced with a DAWN digital signal processing (DSP) multi- angle laser light scattering (MALLS) (Wyatt Corp., Santa Barbara, CA) and refractive index (RI) detector for absolute molecular weight determination. GPC was calibrated in accordance to the universal calibration method with narrow range polystyrene standard.
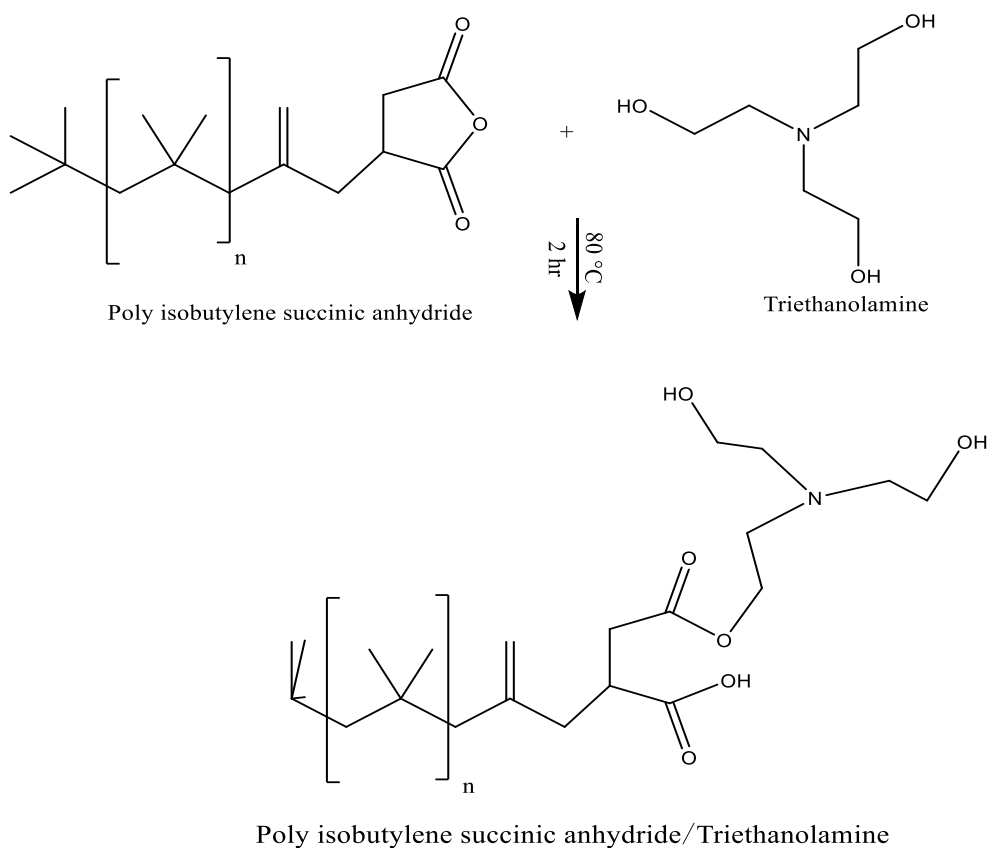
3. RESULTS AND DISCUSSION

3.1. Materials synthesis

PIBSA-1, PIBSA-2, PIBSA/TEA, PIBSA/TEA-1 and -2 were synthesized according to schemes 1 and 2.

Poly isobutylene   +   Maleic Anhydride

250 °C
10 hr

Poly isobutylene succinic anhydride
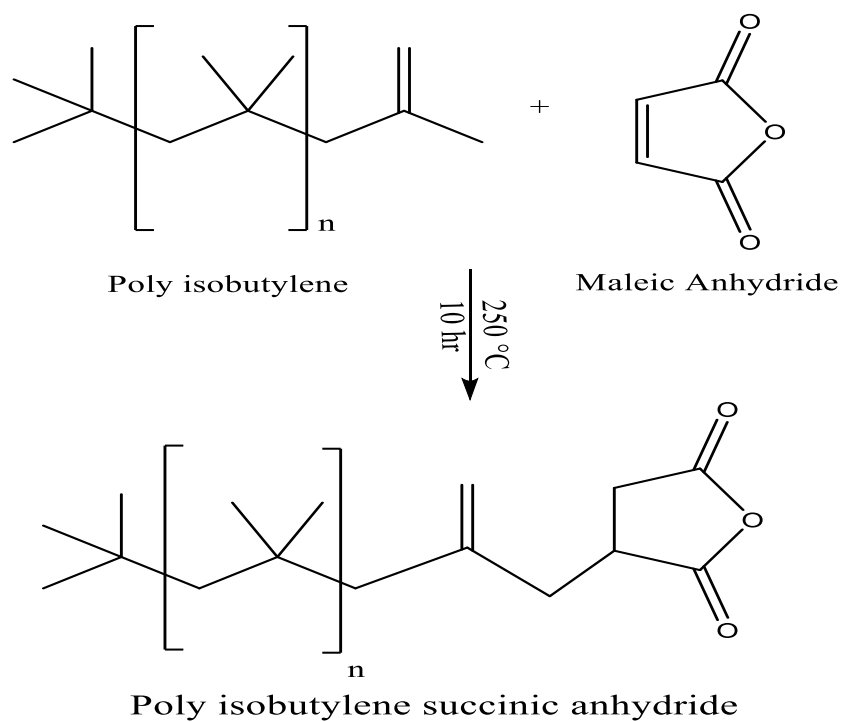
Scheme 1 Typical industrial process for preparation of PIBSA-based surfactants from HR-PIB and Maleic

Poly isobutylene succinic anhydride   +   Triethanolamine

80 °C
2 hr

Poly isobutylene succinic anhydride/Triethanolamine

Scheme 2 Typical industrial process for preparation of PIBSA/TEA from PIBSA and TEA.
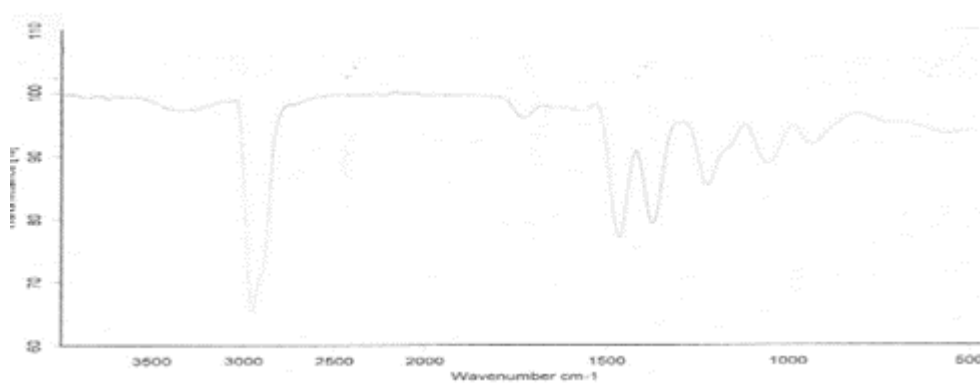
3.2. FTIR spectra
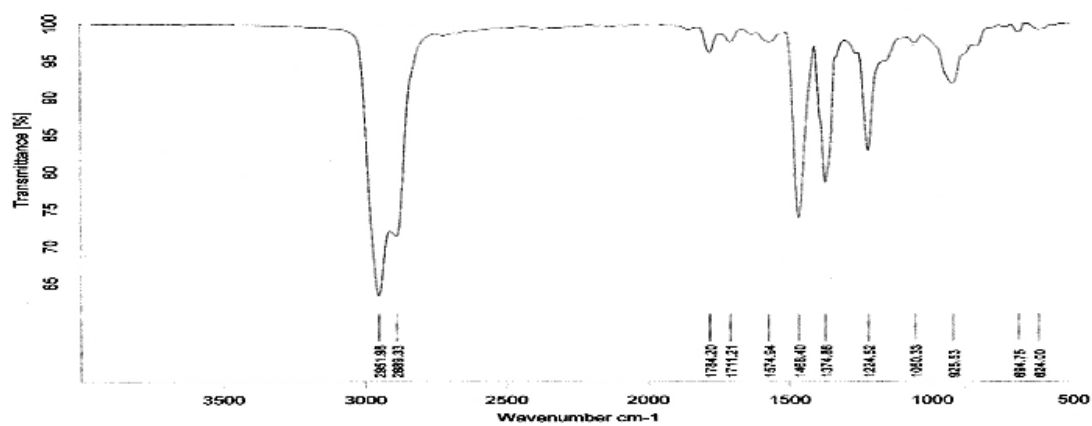
Fig. 1 FTIR spectrum of PIBSA
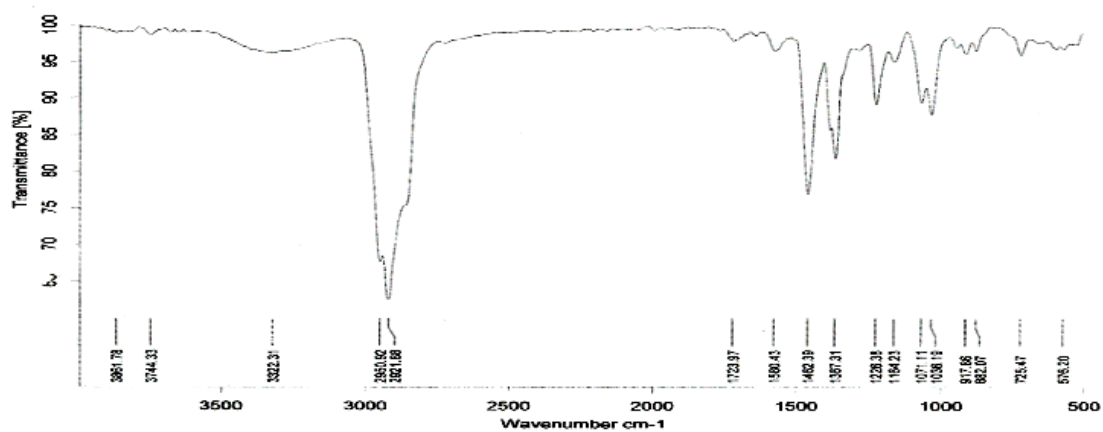

Fig. 2 FTIR spectrum of PIBSA/TEA.

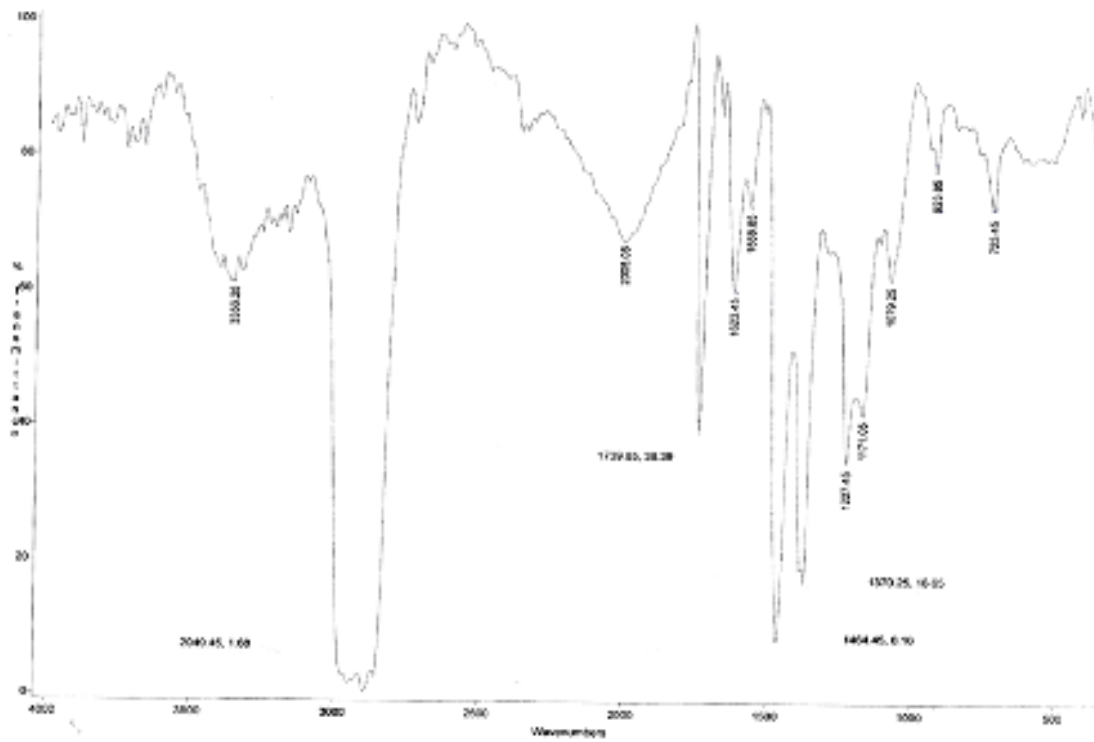**Fig. 3 FTIR spectrum of PIBSA/TEA 1**

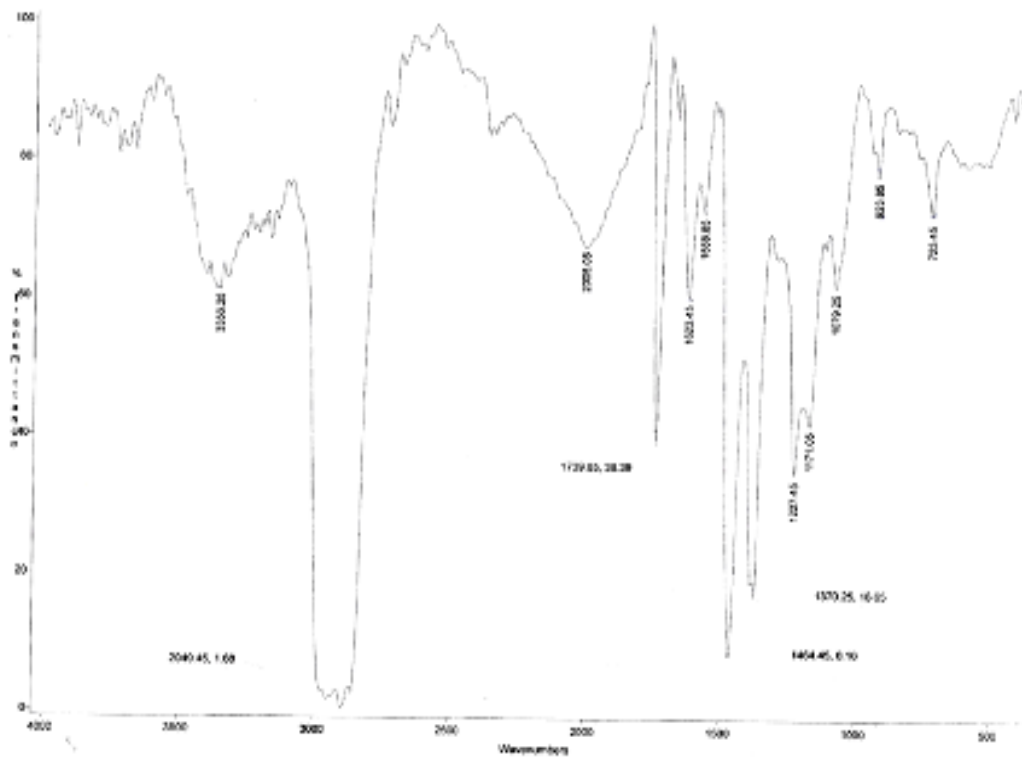**Fig. 4 FTIR spectrum of LZ2735**

Fig. 5 FTIR spectrum of LZ2745

The FT-IR frequencies obtained for the prepared compounds were presented in Table 1.

Table (1) Assignments and comparison of the FTIR frequencies (cm^{-1}) for the various prepared compounds

PIB tail (main chain)	PIBSA 1	PIBSA 2	PIBSA/TEA	PIBSA/TEA 1	PIBSA/TEA 2	LZ2735	LZ2745
Amine N-H str.	-	-	3351	3322.31	3341.88	-	3356.25
CH str.	2951.92	2951.98	2962	2950.92	2950.98	2930.25	2949.45
	2889.35	2889.33	2881	2921.88	2921.64	1924.05	2005.05
CH bend	1468.68	-	1055	-	1071.74	-	-
CH$_3$ swing	1374.79	1374.86	1372	1367.31	1367.63	1377.45	1370.625
CH bend crowded –CH$_2$–	1224.33	1224.52	1225	1226.38	1226.72	1225.65	1227.45
C=C stretch	-	1060.33	-	1462.39	-	1463.85	1464.45
	-	1468.40	1466	917.86	1462.53	-	923.85
C=C bending	928.83	925.53	936	882.07	920.39	-	1623.45
C=C cyclic alkene	-	1574.94	-	1580.43	-	1575.45	1568.85
Head group							
>C=O	1783.48	1784.20	-	-	-	1732.65	1739.85
C=O str. anhydr.	1714.02	1711.21	-	1723.97	-	-	-
C–(CH$_2$) bend	-	-	-	1164.23	1160.19	1172.25	1171.05
Anhydr.	-	-	-	725.47	726.18	-	723.45
N–C	-	624	-	576.20	566.71	-	
C-O-C (main chain)	-	-	-	-	-	1077.45	1079.25
C-OH (main chain)	-	-	-	-	-	1034.25	-

The assignments of FTIR data of PIBSA-1, PIBSA-2, PIBSA/TEA, PIBSA/TEA-1 and PIBSA/TEA-2 in comparison with commercially PIBSA (LZ2735, LZ2745) are shown in Tables 1. The PIBSA-1 and -2 spectral features of the PIB tail (main chain) have shown CH stretch band in the range from 2951–2889 cm^{-1}, CH bend at 1468 cm^{-1}, CH$_3$ swing at 1374 cm^{-1}, CH bend crowded CH$_2$ peaks at 1224 cm^{-1}, C=C stretch at 1060–1468 cm^{-1}, C=C bending peaks at 928 – 925 cm^{-1}, C=C cyclic alkene at 1574 cm^{-1}. Whilst the head group of succinic anhydride C=O stretch vibration appeared at 1714, 1711, 1783 and1784 cm^{-1}.

The PIBSA/TEA is characterized by a CH stretch band in the range from 2962– 2881 cm^{-1}, CH bend at 1055 cm^{-1}, CH$_3$ swing at 13742 cm^{-1}, CH bend crowded CH$_2$ peaks at 1225 cm^{-1}, C=C stretch at 1466 cm^{-1}, C=C bending peak at 936 cm^{-1} and amine moiety at 3351 cm^{-1}.

PIBSA/TEA-1 and -2 have displayed CH stretch band in the range from 2950 - 2921 cm^{-1}, CH bend at 1071 cm^{-1}, CH$_3$ swing at 1367 cm^{-1}, CH bend crowded CH$_2$ peaks at 1226 cm^{-1}, C=C stretch at 1462 cm^{-1}, C=C bending peak at 920 cm^{-1}. The amine group was located at 3341 cm^{-1}. The C-(CH$_2$) bend vibration was noticed at 1160 cm^{-1} and the anhydride vibration at 726 cm^{-1}.

The commercial LZ2735 sample was indicated by the CH stretch at 2930 and 1924 cm^{-1}, CH$_3$ swing at 1377cm^{-1}, CH bend crowded CH$_2$ peaks at 1225 cm^{-1}, C=C stretch at 1463 cm^{-1}, C=C cyclic alkene at 1575 cm^{-1}, C=O peak at 1732cm^{-1}, C-(CH2) bend at 1172 cm^{-1}, C-O-C (main chain) at 1077 cm^{-1} and C-OH (main chain) at 1034 cm^{-1}. LZ2745 has

shown CH stretch at 2949 and 2005 cm^{-1}, CH$_3$ swing at 1370 cm^{-1}, CH bend crowded CH$_2$ peak at 1227 cm^{-1}, C=C stretch at 1464 cm^{-1}, C=C stretch at 1464 cm^{-1}, C=C bending peak at 923 cm^{-1}. The amine group was absorbed at 3356 cm^{-1}, C=C cyclic alkene at 1568 cm^{-1}, C=O group at 1739 cm^{-1}, C-(CH$_2$) moiety at 1171 cm^{-1}, C-O-C (main chain) at 1079 cm^{-1} and the anhydride group at 723 cm^{-1} .

These peaks at 1714, 1711 cm^{-1} and 1783, 1784 cm^{-1} represented the carbonyl group of succinic anhydride, respectively. The disappearance of the peak at 1711 cm^{-1} in the bottom FT-IR spectrum illustrates that all the

3.2.1 ^{1}H NMR

maleic anhydride (MA) is converted to succinic anhydride after PIBSA was subject to the process described in Scheme 1. LZ27435 (SMO/tetra methyl ammonium hydroxide) and LZ2745 (PIBSA/SMO)[14,15], were characterized by carbonyl stretching of the succinic carboxylic group. PIBSA/TEA is identified by amine which it is absorbed at 3351, 3322, 3341 and 3356, respectively.

3.2 (^{1}H, ^{13}C) NMR spectra

^{1}H- and ^{13}C-NMR data indicated fractionation into components for PIBSA-1, -2, PIBSA/TEA-1, -2 along with LZ2735 and LZ2745 as presented in Tables 2 and 3.
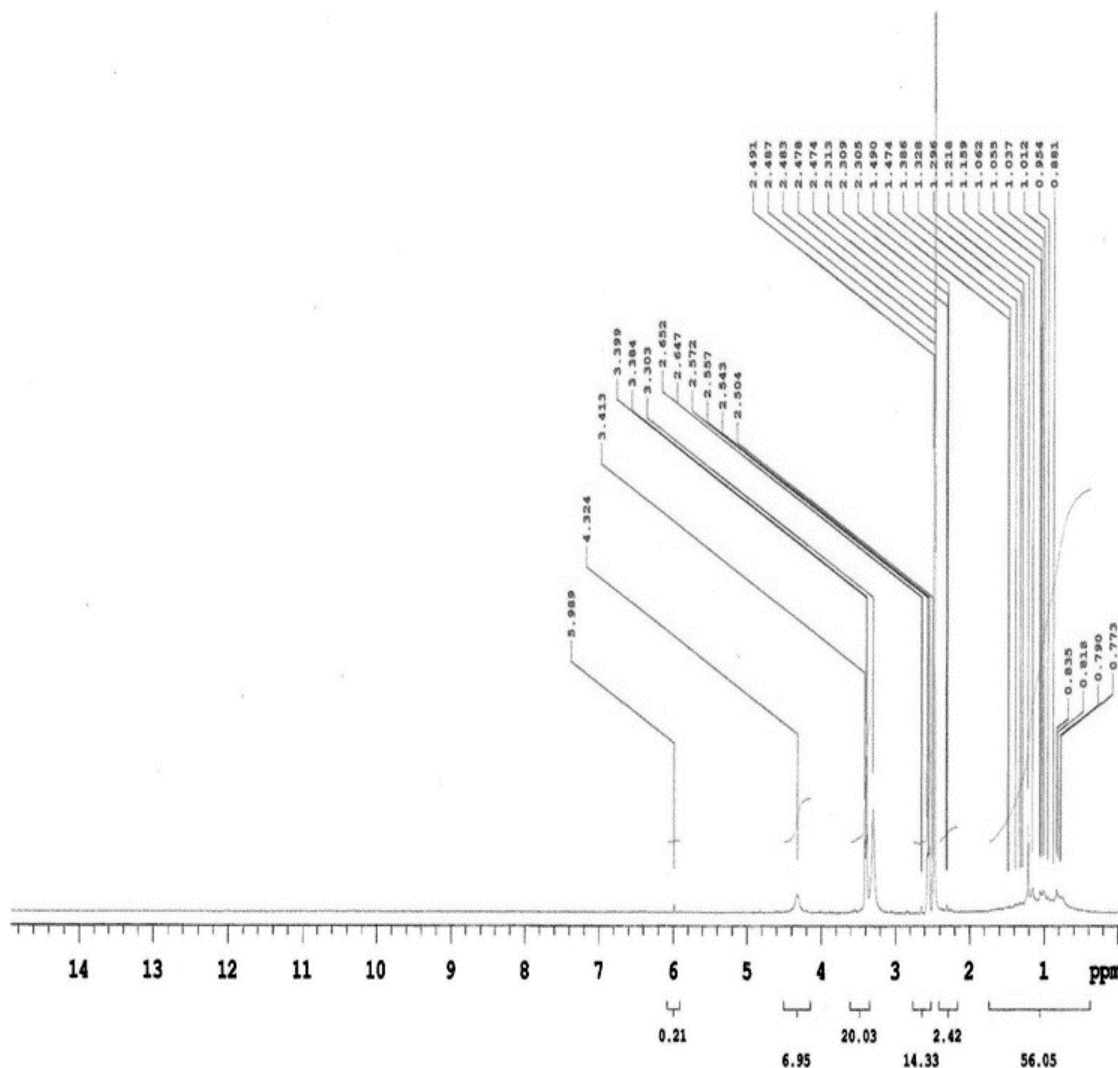

Fig. 6 ^{1}H-NMR spectrum (400MHz, DMSO) of PIBSA

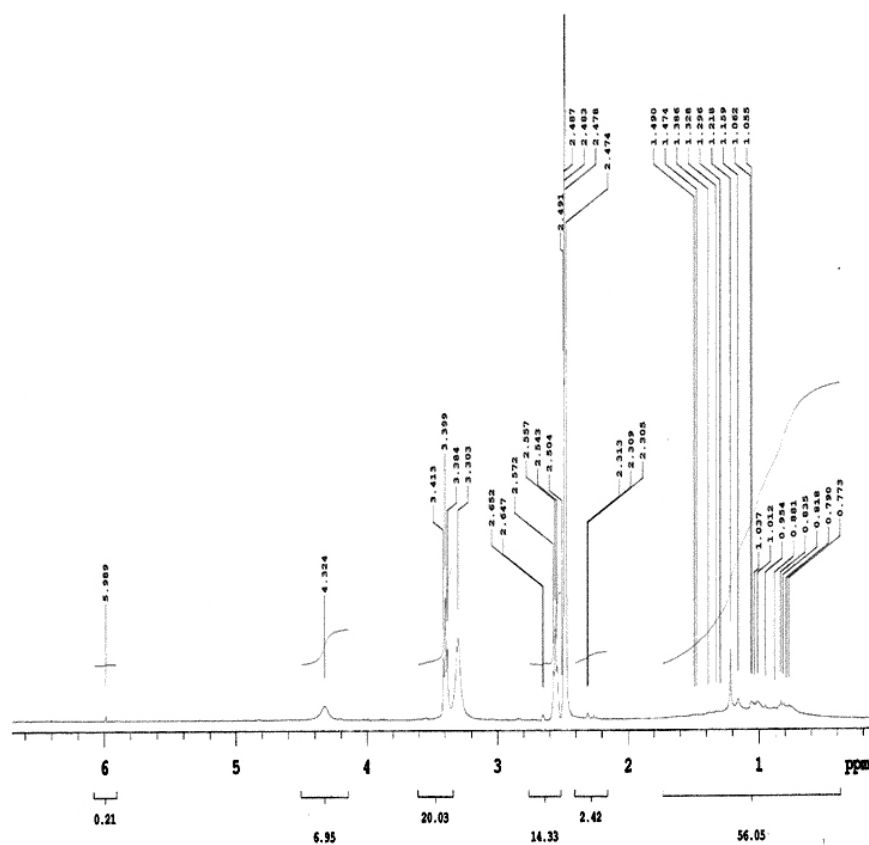RABIE S. FARAG, *et al.*,

Fig. 7 ¹H-NMR spectrum (400MHz, DMSO, D2O) of PIBSA/TEA 1
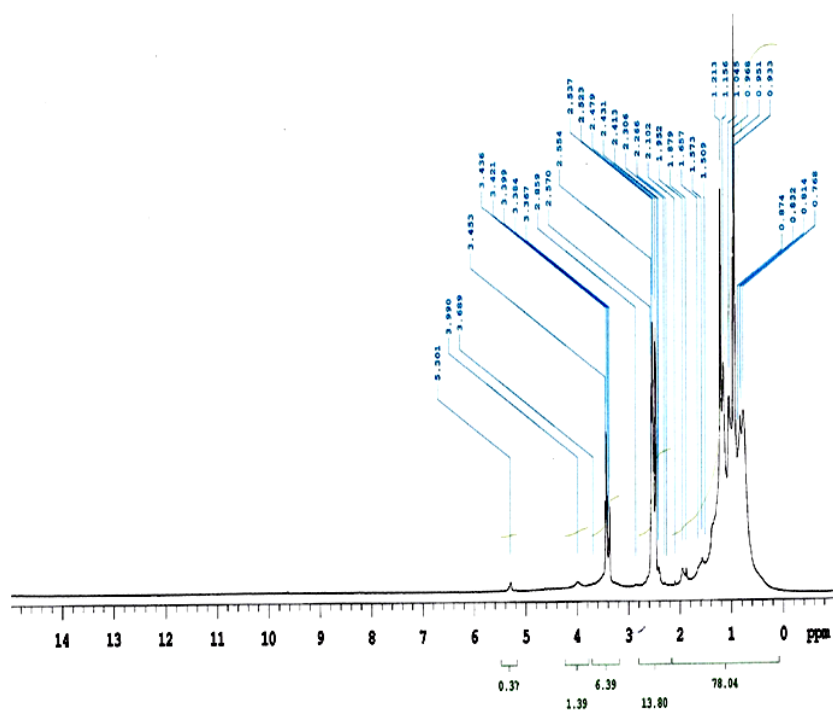
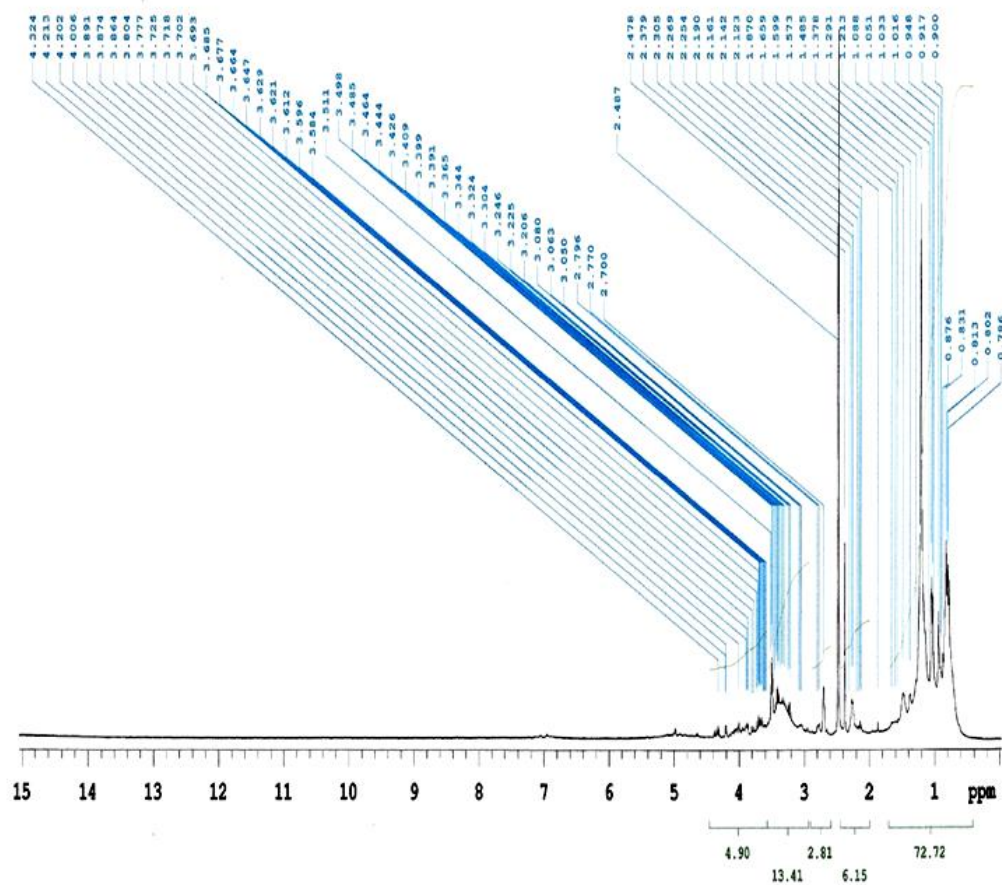**Fig. 8 ¹H-NMR spectrum (400MHz, DMSO, D2O) of LZ273 5**

Fig. 9 1H-NMR spectrum (400MHz, DMSO, D2O) of LZ2745

Table (2) ¹H NMR of PIBSA-1, -2, PIBSA/ TEA-1, -2, LZ 2735 and LZ 2745.

	PIBSA 1	PIBSA 2	PIBSA/TEA 1	PIBSA/TEA2	LZ2735	LZ2745
CH₃	0.835		0.773, 0.790, 0.818, 0.835, 0.881, 0.954, 1.065.	0.818, 0.835, 0.954, 1.011.	0.768,0.814 0.832,0.874 0.933,0.951 0.968,1.045	0.786,0.802 0.876,0.900 0.941,1.016 1.033,1.088
CH₂	1.218	1.218	1.328	1.218, 1.326	1.213	1.21,1.37
CH			1.47, 1.159	1.159	1.156,1.509 1.573,1.657	1.573,1.659 1.870
OH Alcohol			3.303 - 3.399 – 4.24	3.389 , 4.24	3.36, 3.42 3.68 ,3.99	3.34,3.51 3.69,3.87
Amide				5.989		
Vinylic conjugated C=CHR	6.229	6.238			5.3	
Aromatic ring	7.453	7.452				
C=C-CH₃					1.879,1.952	
RC=OCH₃					2.102,2.266 2.306	2.123,2.254 2.305,2.379
R₂N-CH₃						2.70,3.05

The ^{1}H NMR of PIBSA-1 and -2 reported chemical shifts (δ_H) for CH$_2$ 0.835; CH$_3$ 1.218; C=CHR 6.238, and CH$_3$-C-C-G (G = Ring) 7.453 cycle of succinic anhydride.

The PIBSA/TEA-1 sample showed δ_H at 0.773, 0.790, 0.818, 0.835, 0.881, 0.954 ,1.065, 1.159 ppm were assigned to CH$_3$, while CH$_2$ and CH appeared at 1.328 and 1.47 ppm, respectively. The hydroxyl group proton was at 3.303-4.24 ppm, and that at 5.989 ppm was referred to CHR. PIBSA/TEA-2 gave δ_H at 0.818, 0.835, 0.954, 1.011, 1.159, 1.218 ppm which indicated the presence of CH$_3$. CH$_2$ appeared at 1.326 ppm and the hydroxyl proton at 3.389 and 4.24 ppm.

The LZ 2735 sample displayed CH$_3$ at δ_H = 0.768, 0.814, 0.968, 1.045 ppm, CH$_2$ at 1.213 ppm, CH at 1.156, 1.505, 1.571, 1.657 ppm, OH of the hydroxyl group proton at 3.36, 3.42, 3.68, 3.99 ppm and RC=OCH$_3$ at 2.102, 2.266, 2.306 ppm. Interestingly, the C=C-CH$_3$ moiety in LZ 2735 appeared at δ_H = 1,87 and 1095 ppm. LZ 2745 has shown the same chemical shifts except the latter feature was absent, although a newly R$_2$N-CH$_3$ moiety appeared at δ_H = 2.70 and 3.05 ppm.

3.2.2 ^{13}C NMR
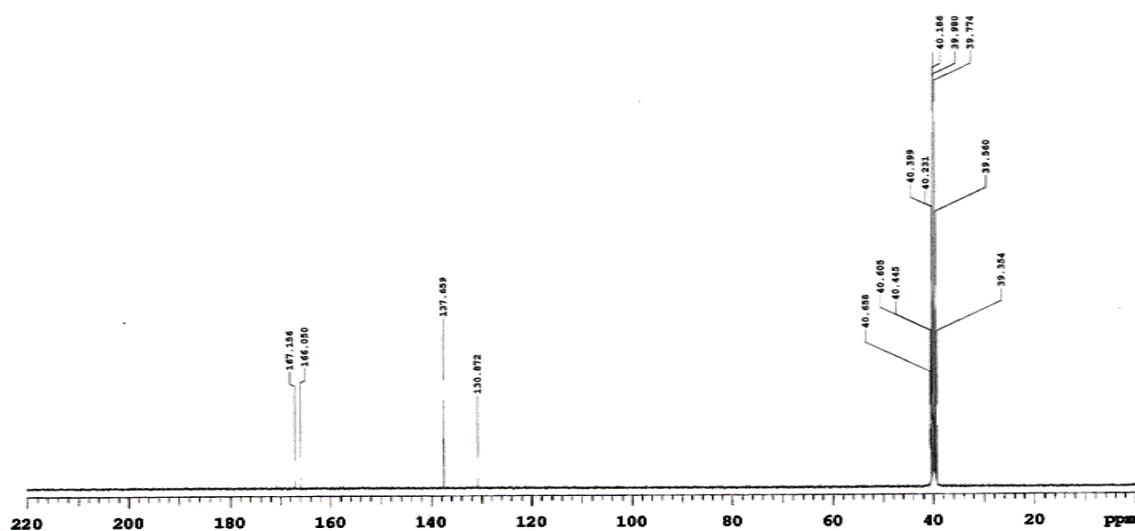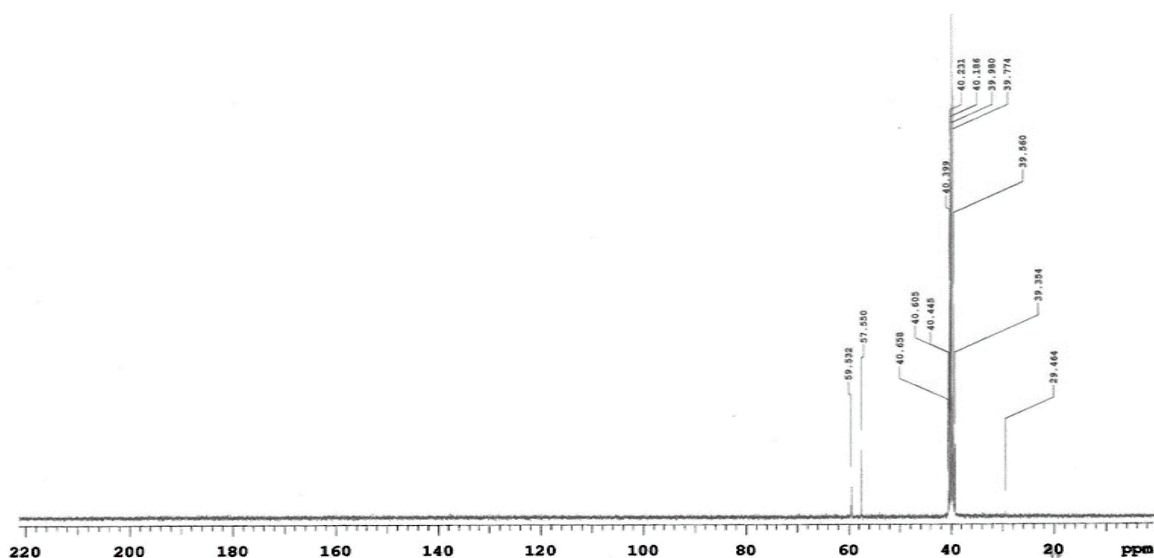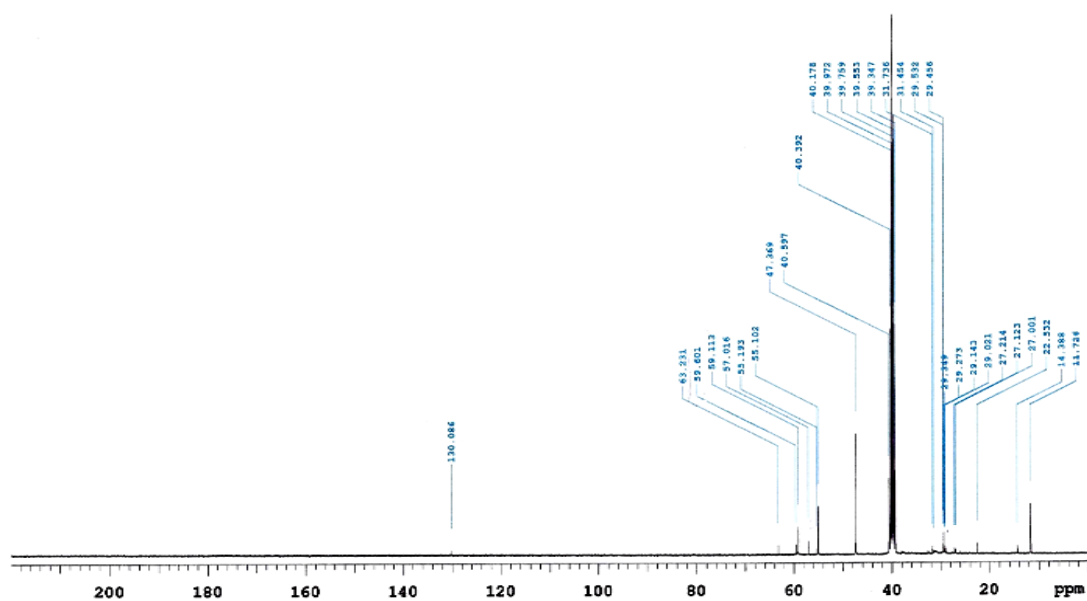

Fig. 10   ^{13}C-NMR spectrum (400MHz, DMSO) of PIBSA

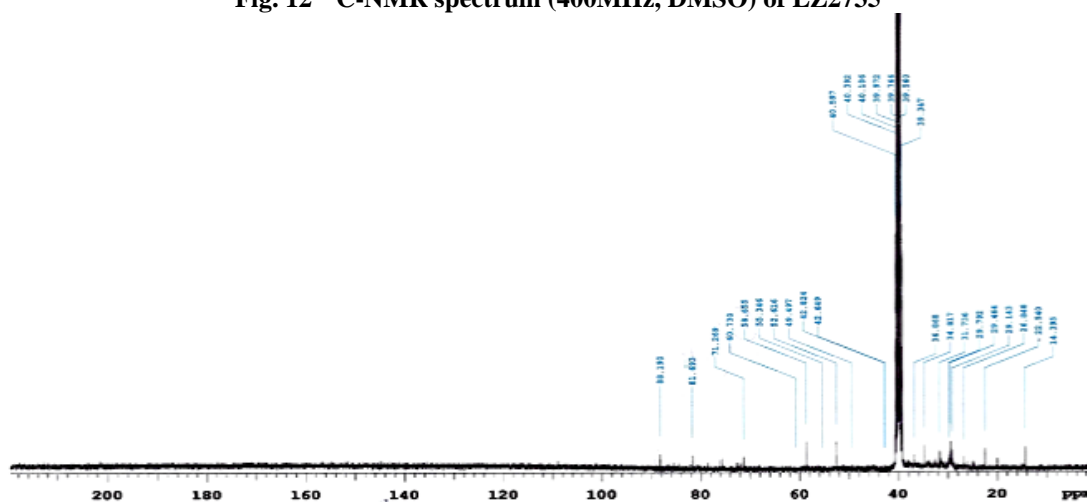

Fig. 11 ^{13}C-NMR spectrum (400MHz, DMSO) of PIBSA/TEA 1

Fig. 12 ^{13}C-NMR spectrum (400MHz, DMSO) of LZ2735

Fig. 13 ^{13}C-NMR spectrum (400MHz, DMSO) of LZ2745

Table (3) ^{13}C NMR for PIBSA 1, 2, PIBSA/TEA-1, -2, LZ 2735 and LZ 2745.

	PIBSA 1	PIBSA 2	PIBSA/TEA 1	PIBSA/TEA2	LZ2735	LZ2745
Cin Aromatic rings C=C (in Alkenes)	137.659	130.872 137.659				
C=O esters		166.050 167.156				
RCH$_2$O			57.550 59.532	57.603 59.639	55.10,57.01 59.60,63.23	52.61,58.65 60.73,81.69
R-CH$_3$					11.72-31.73	14.39-36.86
R$_2$CH$_2$					47.369	42.64,49.49
R-CHO					193.571	
R-COOH						186.00

The ^{13}C-NMR chemical shift values for PIBSA-1 reported δ_C at 137.659 ppm due to C in aromatic ring C=C, while PIBSA-2 presented with one set of carbonyl peaks of the succinic anhydride at 130.872 and 137.659ppm. The C=O ester indicated δ_C at 166.050 and 167.156 ppm for both PIBSA-1 and -2 samples. The PIBSA/TEA-1 and -2 revealed δ_C at 57.550 and 59.532 ppm, and 57.60 and 59.37 ppm, respectively, assigned to RCH_2O.

LZ 2735 illustrated RCH_2O at δ_C = 55.10, 57.01, 59.60 and 63.23 ppm, RCH_3 at 11.72 and 31.73 ppm, R_2CH_2 at 47.69 ppm and RCHO at 193.57 ppm. LZ 2745 showed same characteristics except the presence of RCHO moiety in the former only, although the RCOOH one was newly appeared for the latter only at 186 ppm.
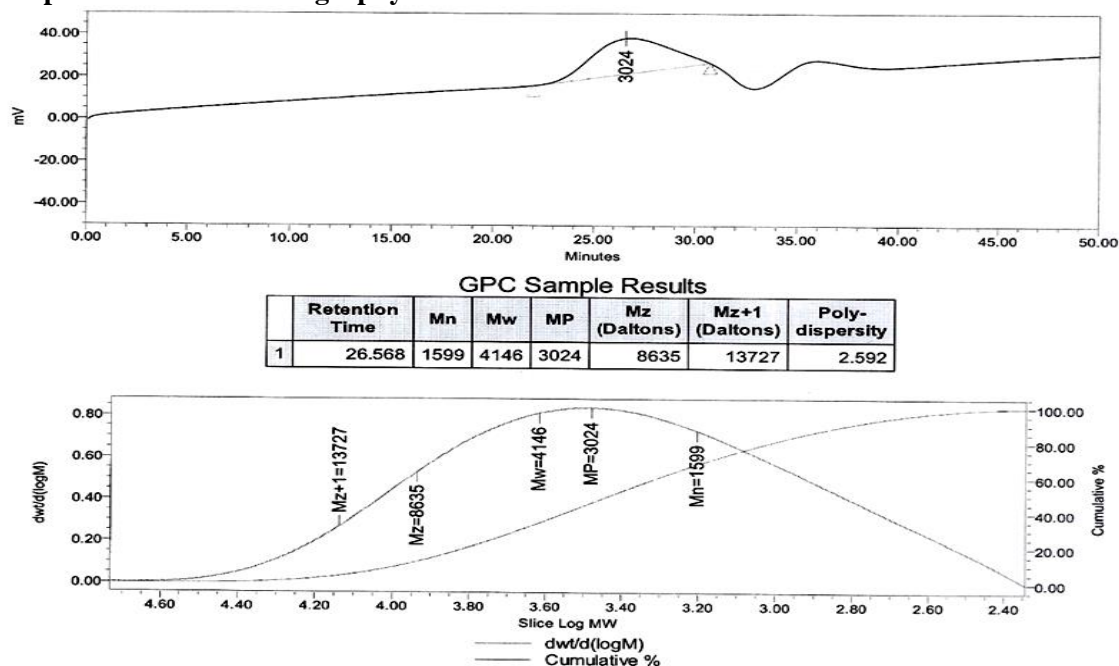
3.3 Gel permeation chromatography

GPC Sample Results

	Retention Time	Mn	Mw	MP	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	26.568	1599	4146	3024	8635	13727	2.592

Fig. 14 GPC of PIBSA

GPC Sample Results

	Retention Time	Mn	Mw	MP	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	26.217	3657	4946	4205	6536	8168	1.352
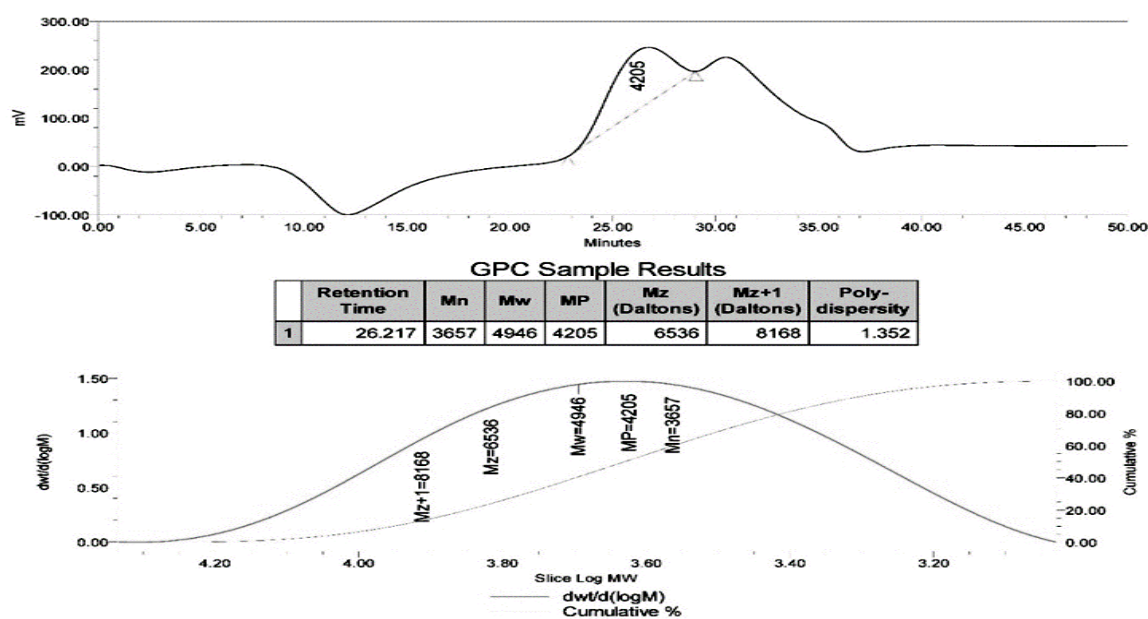
Fig. 15 GPC of PIBSA/TEA 1
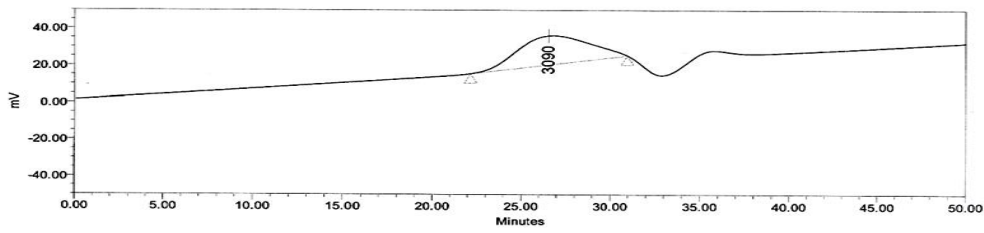
GPC Sample Results

	Retention Time	Mn	Mw	MP	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	26.533	1469	4263	3090	9481	15464	2.901
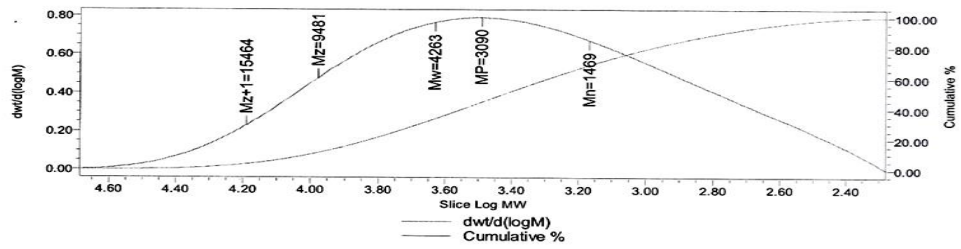
Fig. 16 GPC of PIBSA/TEA
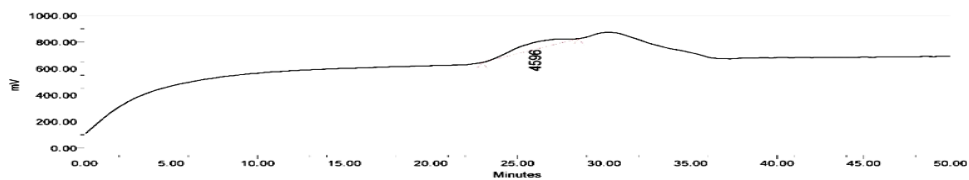
GPC Sample Results

	Retention Time	Mn	Mw	MP	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	26.033	4134	5398	4596	6903	8391	1.306
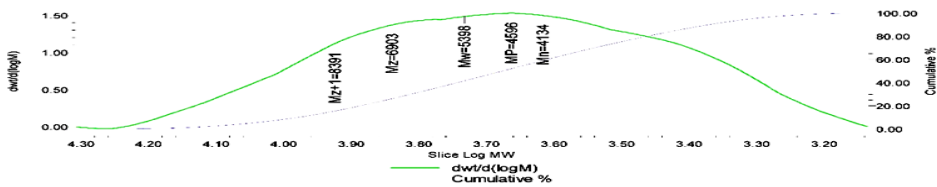
Fig. 17 GPC of Emulsifier LZ2735
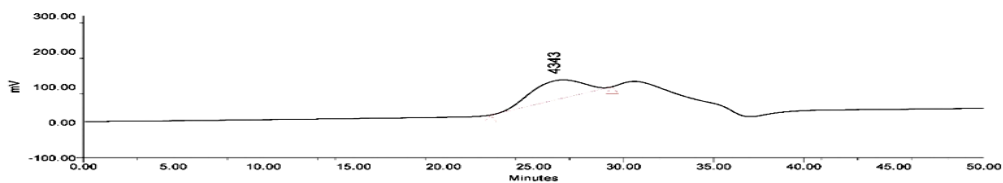
GPC Sample Results

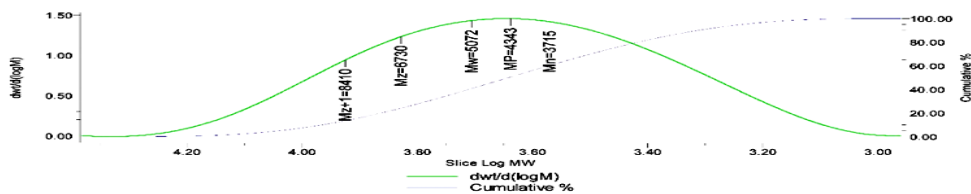|	Retention Time	Mn	Mw	MP	Mz (Daltons)	Mz+1 (Daltons)	Poly-dispersity
1	26.150	3715	5072	4343	6730	8410	1.365

Fig. 18 GPC of Emulsifier LZ2745

The GPC data are given in table (4).

Table 4 Gel permeation chromatography (GPC).

Comparison Compounds	Retention Time	M_n	M_w	M_p	M_z (Daltons)	M_{z+1} (Daltons)	Ploy dispersity index = M_w/M_n
PIBSA	26.568	1599	4146	3024	8635	13727	2.592
PIBSA/TEA*	26.533	1469	4263	3090	9481	15464	2.901
PIBSA/TEA1*	26.217	3657	4946	4205	6536	8168	1.352
PIBSA/TEA2*	26.150	3715	5072	4343	6730	8410	1.365
Lubrizol LZ2735	26.167	4042	5087	4308	6356	7660	1.259
Lubrizol LZ2745	26.033	4134	5398	4596	6903	8391	1.306

Run Time = 50 Min, Solvent is THF.

*(PIBSA/TEA is without paraffin oil and PIBSA/TEA1, 2 is mixed with paraffin oil).

The GPC measured the number of average molecular weight (M_n) and weight average molecular weight M_w. Higher average molecular weights: (M_z, M_{z+1}) and M_p= molecular weight of the highest peak. The polydispersity index (polydispersity index = M_w/M_n) was suggestive of presence of a significant amount of higher M_n fully saturated oligomers, likely to result from the second order decomposition reactions of the PIB main chains in several synthetic cycles. The polydispersity index of the prepared PIBSA/TEA improved with adding paraffin oil. Compared with commercial LZ2735 and LZ2745 as indicated in Table (4).

4. APPLICATION

The prepared emulsifiers were tested to selectively absorb some heavy metal ions such as Pb^{2+}. Thus, mixing 0.1g of the PIBSA/TEA emulsifier with of Pb^{2+} (1912 ppm) with stirring, then 5ml portions were withdrawn after 1, 2 and 3 h for analysis by the microwave plasma emission spectroscopy showed that the concentration of Pb^{2+} was reduced by 16%. This indicates that the emulsifier can be used as a membrane for absorption of Pb^{2+} ions form wastewater.

5. CONCLUSION

This study deals with novel prepared polyisobutylene succinic anhydride/ Triethanolamine (PIBSA/TEA) emulsifiers which are important to many industries. From previous studies, the prepared emulsifiers were used as emulsion explosives, detergents, cosmetics, paintings. Also, the emulsifiers can be used as membrane from selectively absorb Pb^{2+} from wastewater.

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الملخص العربى:

المستحلبات تعـرف بأهميتهـا فـى كثيـر مـن الصـناعات وبـالأخص مـادة بـولى أيزوبيـوتيلين سكسـينك أنهيدريـد ومشـتقاتها ، التـى تـدخل فـى صـناعات مستحضـرات التجميـل والمنظفـات والزيـوت البتروليـة والمفرقعـات المسـتحلبة حيـث تسـتخدم كمـواد رابطـة بـين المحاليـل والمواد البترولية وتستخدم فى زيادة ثبـات المـواد وحفظ صـلاحيتها ، وفـى هـذه الدراسـة تـم تحضـير مـادة بـولى أيزوبيـوتيلين سكسـينك أنهيدريـد / ثلاثـى إيثـانول أمـين بنسـب موليـة مختلفـة مـع عمـل دراسـات تحليلـة حـول المركبـات لإثباتهـا ومقارنتهـا مقارنـة تحليلـة مـع بعـض المستحلبات المنشورة ، وقد استخدم المستحلب كمرشح لتنقية المياه من الرصاص .