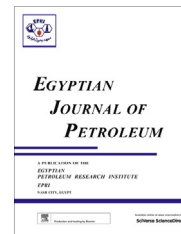




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FULL LENGTH ARTICLE

Synthesis and characterization of high molecular weight hydrophobically modified polyacrylamide nanolatexes using novel nonionic polymerizable surfactants

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Abstract In this article, nine hydrophobically modified polyacrylamides (HM-PAM) nanolatexes, were synthesized by copolymerizing the acrylamide monomer and novel polymerizable surfactants (surfmers). The reaction was carried out by inverse microemulsion copolymerization technique. The copolymerization was initiated by redox initiators composed of potassium peroxodisulphate and sodium bisulfite. The emulsion was stabilized using mixed tween 85 and span 80 as nonionic emulsifiers. The prepared HM-PAMs were classified into three groups according to the surfmers used in the copolymerization. The chemical structures of the prepared HM-PAMs were confirmed by FT-IR, ¹H NMR and ¹³C NMR. The thermal properties were estimated with the thermal gravimetric analysis (TGA). The size and morphology of the prepared latexes were investigated by the dynamic light scattering (DLS) and the High Resolution Transmission Electron Microscope (HRTEM). Finally, the molecular weights of the prepared copolymers were determined by the GPC and the viscosity average molecular weight method. They were situated between 1.58×10^6 and 0.89×10^6 .

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1. Introduction

The water-soluble hydrophobically modified polyacrylamide (HMPAM) has been extensively investigated in the last thirty years. Research interest stems mainly from two areas: solutions and hydrogels. Interest in HMPAM solutions has arisen primarily from controlling viscosity at different shear rates (rheology) in many technologies. A useful strategy for enhanced control of rheology in these systems takes advantage of HMPAM which build viscosity through transient polymer association [1]. Therefore, these systems have received a great

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deal of attention due to their unique rheological characteristics and applications, such as cosmetics, coatings, drilling fluids, and chemically enhanced oil recovery [1–9]. HMPAM consists of a water-soluble polymer containing a small number of hydrophobic groups. Over the years, several classes of associative polymers have been developed: depending on the method of synthesis, the hydrophobes can be end-attached (telechelic polymers) or distributed either statistically or as small blocks in the hydrophilic polymeric backbone (multisticker polymers). They are usually obtained either by chemical modification of a precursor polymer or by free-radical copolymerization of the appropriate monomers. However, a major drawback in the latter process comes from the insolubility of the hydrophobic co-monomer in water. This problem can be overcome by carrying out the polymerization reaction in a mixture of solvents where both monomers are soluble [5,10–12] or by using a micellar polymerization technique in which the presence of a surfactant in the micellar state ensures solubilization of the hydrophobe in the aqueous medium [2,7,13]. The latter process was shown to lead to multiblock copolymers in which the hydrophobic blocks are of tunable length and number, according to the experimental conditions [3,5,7,14–17]. The design of such materials with controllable rheological properties is quite attractive, although the low solid contents achieved at full conversion (a few wt%) may restrict their potential applications.

However, polymerization reactions in inverse emulsions or microemulsions are choice methods for the synthesis of high molecular weight, water-soluble polymers in the form of latexes, that is, water-swollen polymer particles dispersed in an organic, continuous phase [18–20]. The advantages of the techniques are the high solid contents (25–35 wt%) with low viscosities of the dispersions, good handling, and storage facilities. The microemulsions are isotropic, transparent or translucent, and thermodynamically stable multicomponent fluids, normally composed of an aqueous component, an oily component, an amphiphile as surfactant, and frequently a co-surfactant (usually an alcohol of intermediate chain length). The unique properties of the microemulsions such as lower viscosity, greater stability and transparency due to uniformly dispersed smaller droplets have made them attractive media for polymerization [21–25]. Since 1980s, a new polymerization method was developed: inverse microemulsion polymerization and it has become an attractive way to produce high molecular weight water-soluble polymers. Therefore, inverse microemulsion polymerization has recently attracted considerable attention and become an increasingly growing field of research in water-soluble polymer synthesis. Up to now many water-soluble polymers have been prepared by inverse microemulsion polymerizations, most of them are AM-based ones. Some investigators [26] prepared two types of water-soluble polymers through an inverse microemulsion polymerization route: (i) copolymers of acrylamide with sodium-2-acrylamido-2-methylpropanesulfonate and (ii) copolymers of acrylamide and sodium acrylate hydrophobically modified with an amphiphilic monomer, and obtained stable, clear microlatexes of moderate particle size containing up to 25% high molecular weight polymer in the medium.

In the previous work, three groups of nonionic surfmers have been prepared based on alkenylsuccinic anhydride [27]. In this article, these surfmers should be used as co-monomers to prepare three groups of hydrophobically modified poly-

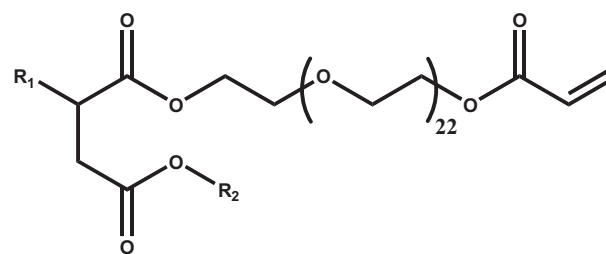


Figure 1 The general formula of the used surfmers.

acrylamides by inverse microemulsion copolymerization, which initiated by redox system (potassium peroxydisulphate and sodium bisulfite). The microemulsion will be stabilized by tween 85 and span 80 as mixed emulsifiers. The amount of the surfmers in the microemulsion should not be more than 1 mol%.

2. Experimental

2.1. Materials

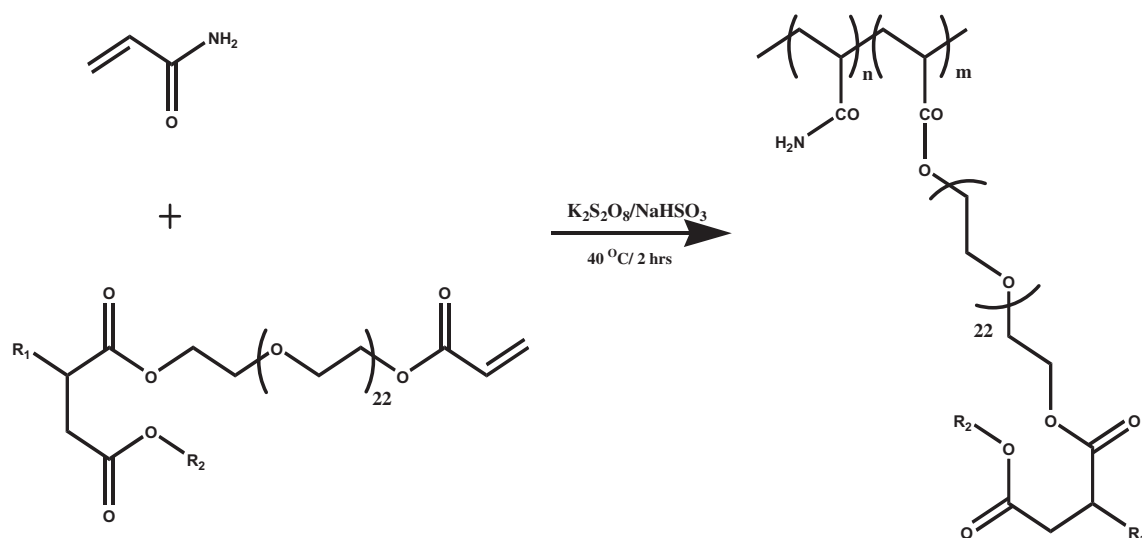
Acrylamide (Am), potassium persulfate (KPS) and sodium bisulfite (NaBiS) were all of analytical grade and used as received without further purification, also solvents; methanol, ethanol and acetone. Kerosene, Sorbitan monooleate (span 80) and polyoxyethylene sorbitan trioleate (Tween 85) were technical grade. The nonionic surfmers based on alkenylsuccinic anhydride were synthesized in the previous work [27]. The general formula of these amphiphilic co-monomers (surfmers) is given in Fig. 1. These nine surfmers were divided into three groups according to the chemical structure. Their abbreviations, molecular weights and HLBs are also presented in Table 1.

2.2. Methods of preparation

The inverse microemulsion copolymerizations for a total recipe of 100 g were carried out in a 250-mL glass reactor fitted with a stirrer, a condenser, a thermometer, and a nitrogen inlet. In all the experiments, the aqueous phase/oil phase ratio and total monomers/water ratio were kept constant as 1/3 and 1/2, respectively. The emulsifier content was 11–13 wt% based on the total recipe and the HLB of the surfmer was used. The aqueous phase [AM (99 mol%) and the proper surfmer (1 mol%)] and the oil phase (kerosene, span 80 and tween 85) were emulsified at 40 °C. The redox initiators KPS ($0.05 \text{ mM} \times 10^{-3}$) and NaBiS ($0.15 \text{ mM} \times 10^{-3}$) were intro-

Table 1 Abbreviations, molecular weights and HLBs of the used surfmers.

Group	Abbreviation	Molecular weight	HLB
Group I	OSA- <i>eo</i> 22Ac (Ia)	1264	15.82
	DDSA- <i>eo</i> 22Ac (Ib)	1320	15.15
	ODSA- <i>eo</i> 22Ac (Ic)	1404	14.24
Group II	O-DDS- <i>eo</i> 22Ac (IIa)	1432	13.96
	TD-DDS- <i>eo</i> 22Ac (IIb)	1516	13.19
	OD-DDS- <i>eo</i> 22Ac (IIc)	1572	12.72
Group III	O-OS- <i>eo</i> 22Ac (IIIa)	1376	14.53
	DD-DDS- <i>eo</i> 22Ac (IIIb)	1488	13.44
	OD-ODS- <i>eo</i> 22Ac (IIIc)	1657	12.07



Scheme 1 The preparation route for the HM-PAMs.

Table 2 Abbreviations, yield, intrinsic viscosities $[\eta]$ and molecular weights of the HM-PAMs and PAM.

Group	Abbreviation	Yield (%)	$[\eta]$ (ml/g)	Molecular weight (g/mol)	
				$M_n \times 10^6$	$M_w \times 10^6$
Group I	PAM-Ia (Poly(AM-co-OSA-eo22Ac))	88	316	1.09	1.29
	PAM-Ib (Poly(AM-co-DDSA-eo22Ac))	90	320	1.11	1.58
	PAM-Ic (Poly(AM-co-ODSA-eo22Ac))	66	336	1.18	1.09
Group II	PAM-IIa (Poly(AM-co-O-DDS-eo22Ac))	77	312	1.07	1.07
	PAM-IIb (Poly(AM-co-TD-DDS-eo22Ac))	66	286	0.95	0.93
	PAM-IIc (Poly(AM-co-OD-DDS-eo22Ac))	67	282	0.93	0.98
Group III	PAM-IIIa (Poly(AM-co-O-OS-eo22Ac))	75	288	0.96	0.95
	PAM-IIIb (Poly(AM-co-DD-DDS-eo22Ac))	80	296	1.01	1.22
	PAM-IIIc (Poly(AM-co-OD-ODS-eo22Ac))	65	293	0.99	0.89
–	PAM (Polyacrylamide)	95	757	3.5	3.44

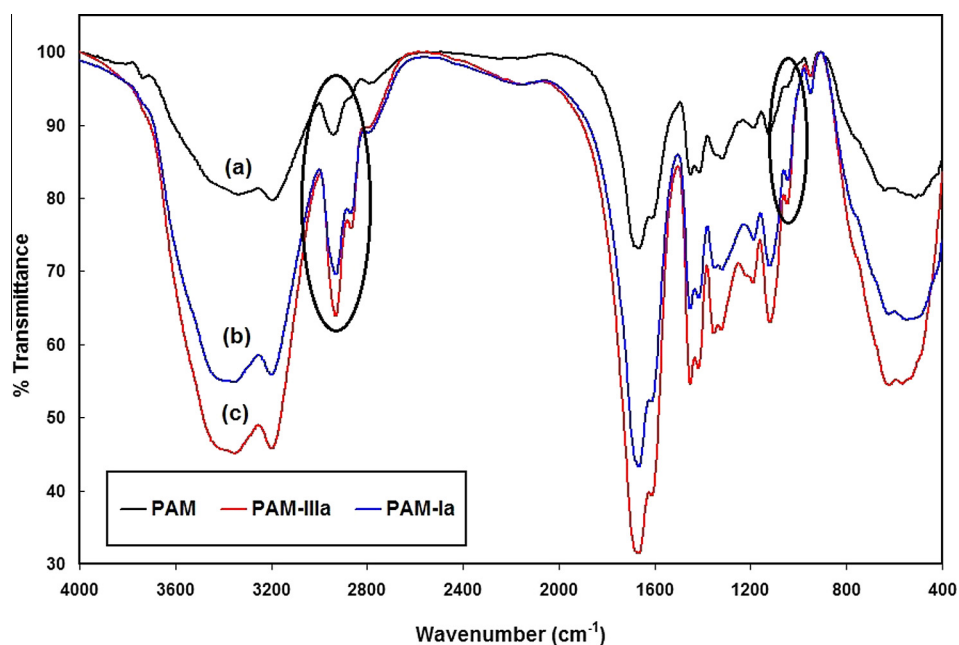


Figure 2 FT-IR Spectra of (a) PAM, (b) PAM-Ia and (c) PAM-IIIa.

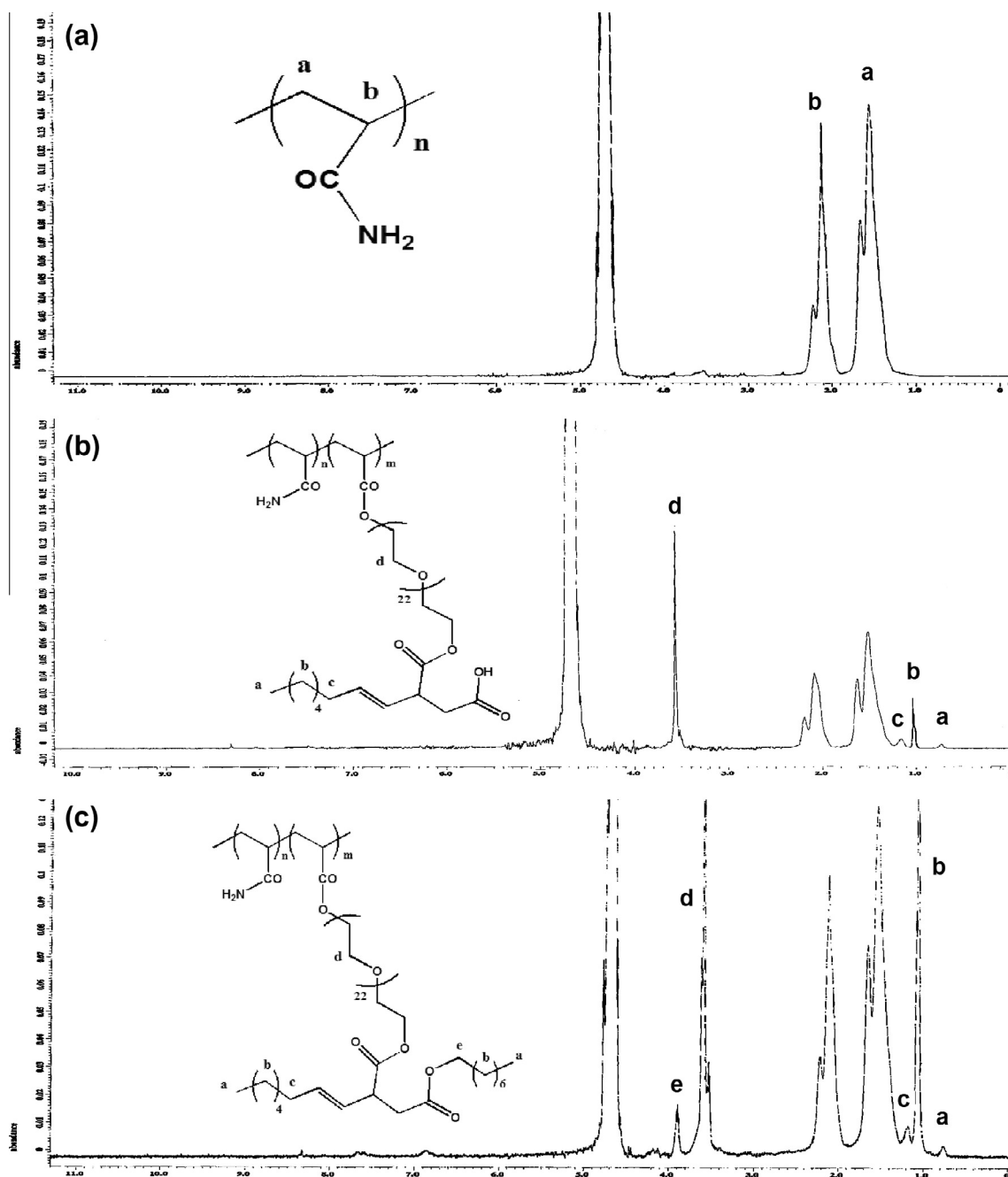


Figure 3 ^1H NMR spectra of (a) PAM, (b) PAM-Ia and (c) PAM-IIIa.

duced to the microemulsions and the reactor was continuously purged with nitrogen from the emulsification to the end of the copolymerization. After 2 h, the resulting product was washed several times with acetone and ethanol then dried at 40 °C to a constant weight. The polyacrylamide (PAM) homopolymer was synthesized with the same method without adding the surfmer [26,28].

2.3. Measurements and analysis

Infrared spectra were performed in KBr disk with a Nicolet FTIR IS-10 spectrometer (Waltham, MA, USA). ^1H and ^{13}C NMR spectra were recorded on a Bruker EMX spectrometer (Billerica, MA, USA) with D_2O as solvent.

Thermal gravimetric analysis (TGA) was performed on simultaneous DSC-TGA Instrument model Q 600 SDT (USA).

The morphology of the copolymers was observed through high resolution transmission electron microscopy. The TEM images have been taken through a JEM-100CXII type. The droplets and particle sizes of the prepared inverse microemulsions before and after copolymerization were determined using dynamic light scattering equipment (Zetasizer Nano-ZS, Malvern, UK) at 30 °C.

The molecular weights of the prepared HM-PAMs were determined by two methods; the first, the polymers were expressed relatively with their intrinsic viscosities $[\eta]$. They were determined using capillary viscometers (Cannon-Fenske) with

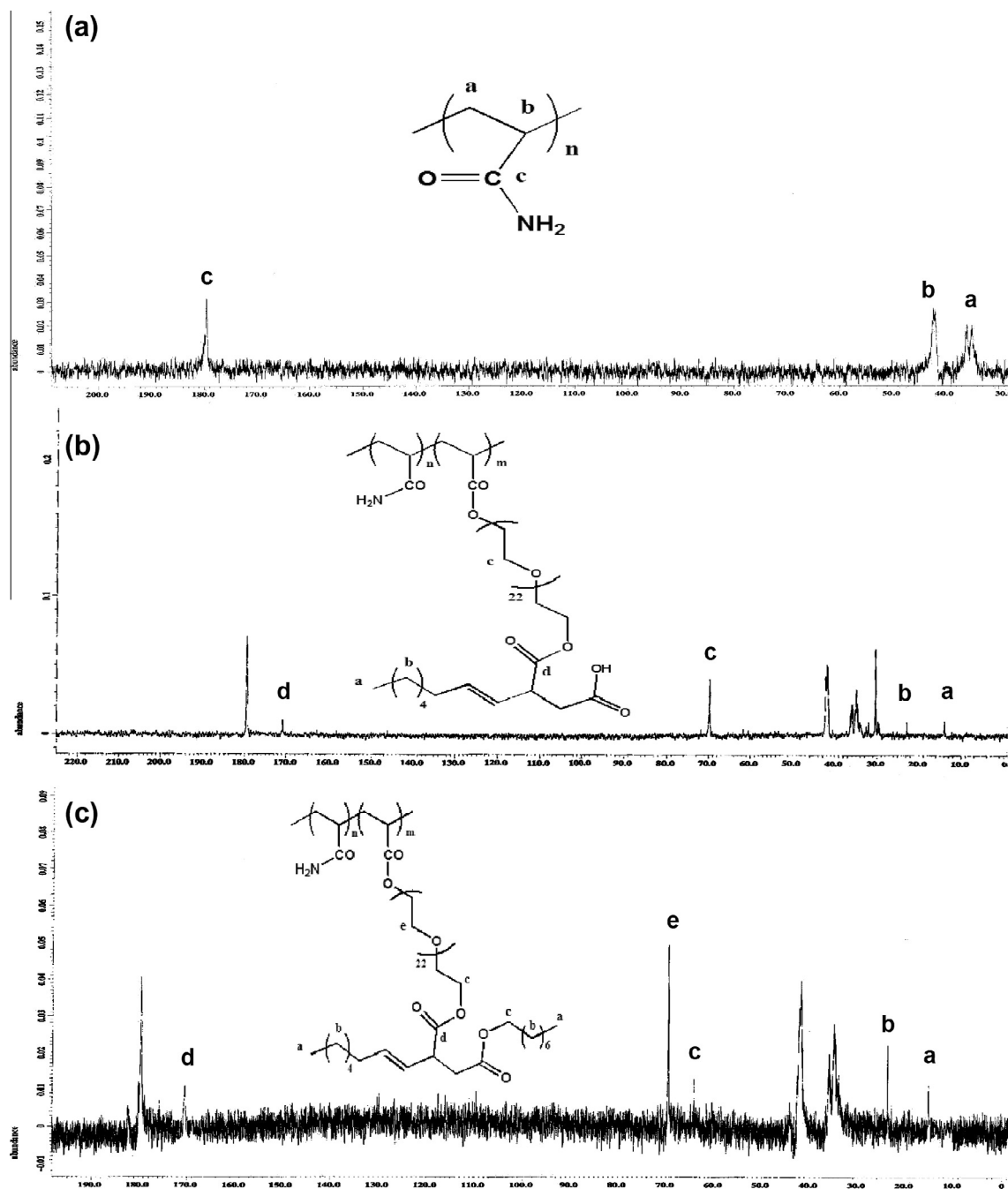


Figure 4 ^{13}C NMR spectra of (a) PAM, (b) PAM-Ia and (c) PAM-IIIa.

0.1 mol/L NaCl solution as solvent and the concentrations of the polymers were in the range of 0.003–0.004 g/ml. The measurements were kept at 25 ± 0.1 °C. The intrinsic viscosities $[\eta]$ of the HM-PAMs were obtained with the dilution extrapolation method. However, it should be pointed out that this treatment is only an approximate and coarse method due to the effect of intramolecular hydrophobic interaction on the determination of viscosity-average molecular weight for the HM-PAMs [29], but it is inferred that the shear rate imposed in the capillary does not affect the viscosity data [5]. The second, using gel permeation chromatography (GPC) instrument (Waters Co., USA). GPC analysis was performed using

0.1 M NaNO_3 aqueous solutions as the eluent and molecular weights of the copolymers were calibrated with Polyethylene glycol standard samples [30].

3. Results and discussions

3.1. Chemical structure justification

The hydrophobically modified polyacrylamides (HM-PAMs) were synthesized according to reaction in Scheme 1. Free radical polymerization of acrylamide only and copolymerization

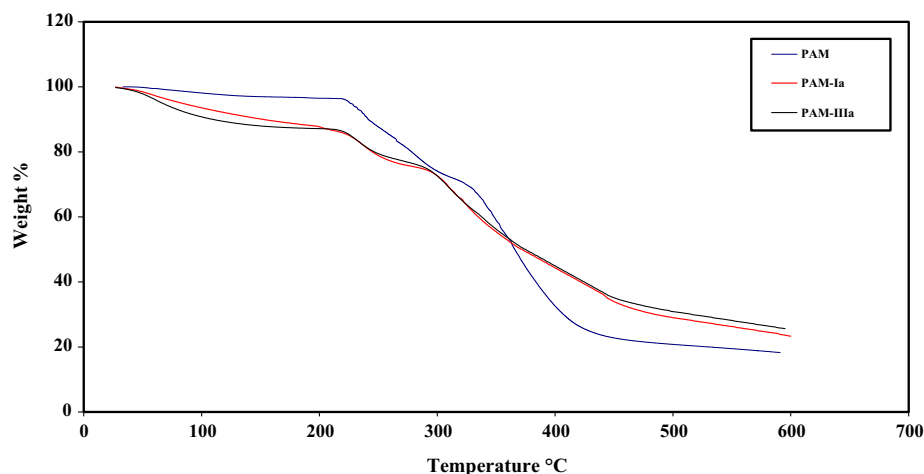


Figure 5 Thermogravimetric curves of PAM, PAM-Ia and PAM-IIIa.

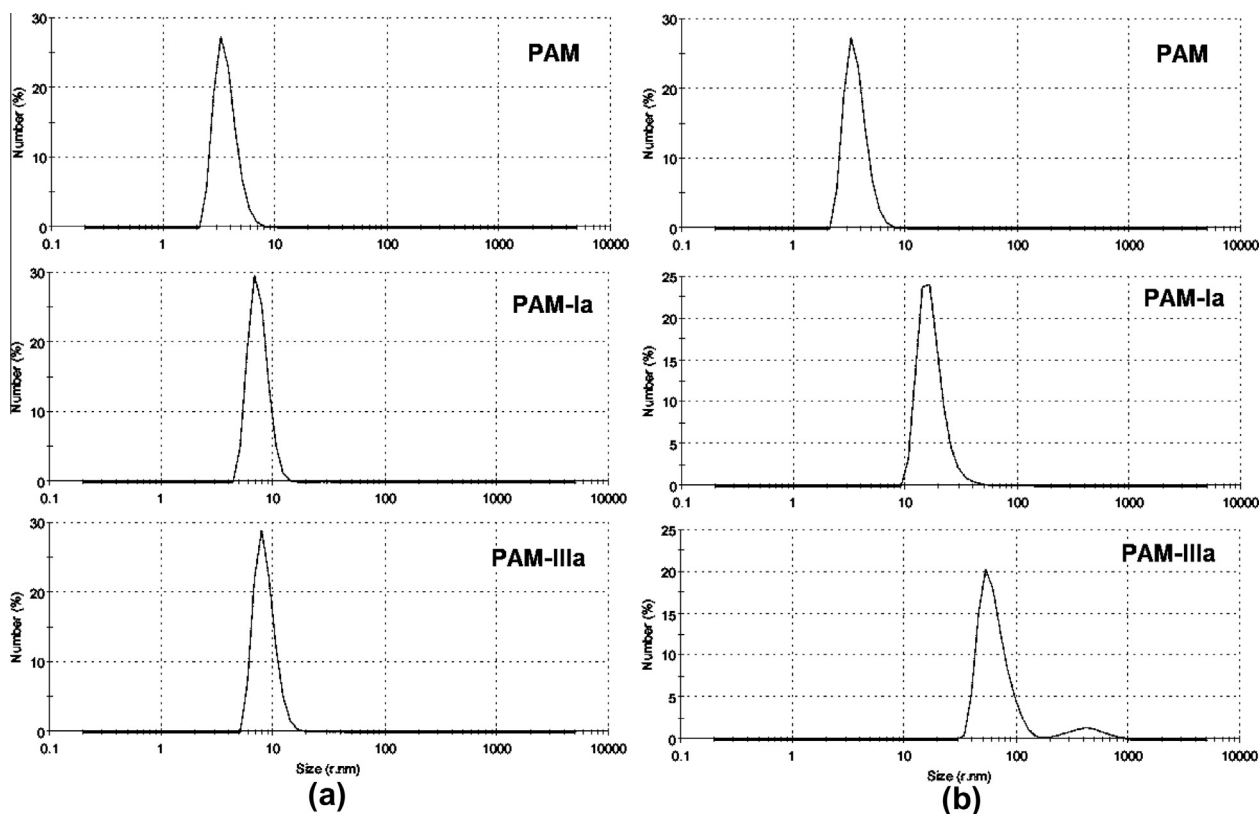


Figure 6 The size distributions of (a) microemulsion droplets before polymerization and (b) latex particles after polymerization for PAM, PAM-Ia and PAM-IIIa.

between acrylamide and each surfmer occurred at low temperature (40 °C) via redox initiation system (KPS and NaBiS). The prepared HM-PAMs were classified into three groups (I, II, III) according to the surfmer groups (group I: single tailed surfmers, group II: asymmetric double tailed surfmers and group III: symmetric double tailed surfmers) as represented in Table 2. The chemical structures of the hydrophobically modified polyacrylamides (HM-PAMs) confirmed by FT-IR spectroscopy for polyacrylamides (PAM), Poly(AM-co-OSA-

eo22Ac) (PAM-Ia) and Poly(AM-co-O-OS-eo22Ac) (PAM-IIIa) as shown in Fig. 2. The FT-IR spectrum (a) of PAM shows characteristic broad bands at around 3400 and 1660 cm^{-1} which can be assigned to $-\text{NH}_2$ and carboxamido groups, respectively. However, The FT-IR spectra (b) and (c) show bands at 2880 and 2940 cm^{-1} which can be assigned to $-\text{CH}_2$ and $-\text{CH}_3$ groups of alkyl chains of the surfmers. Also, the bands at 2940 and 1060 cm^{-1} can be assigned to C–O–C etheral group of the co-monomers. The broad band at

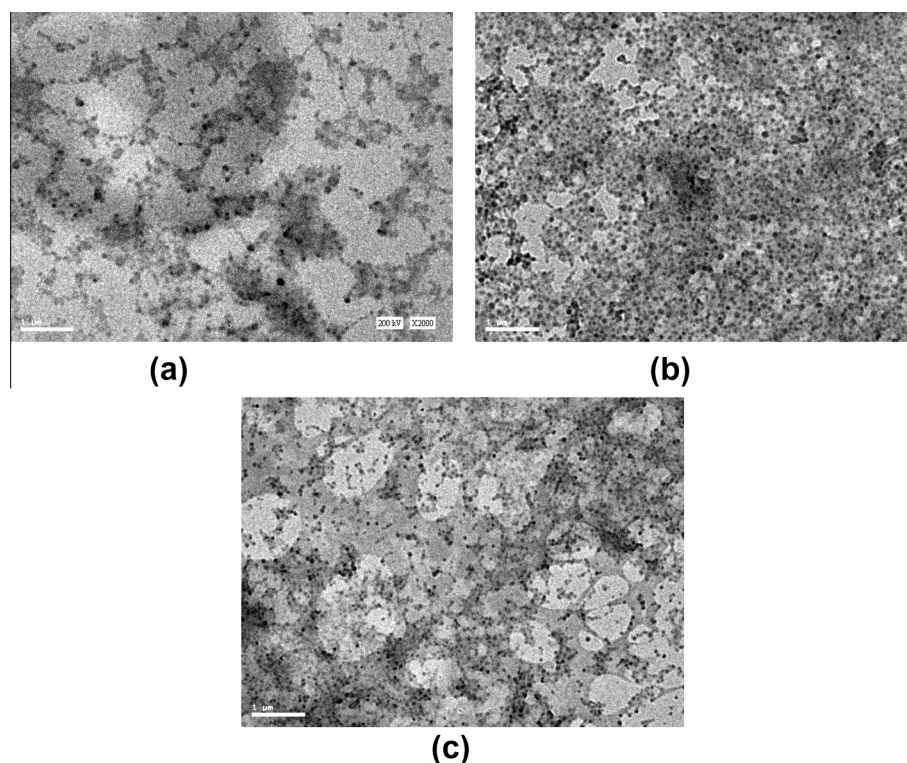


Figure 7 The morphology of the prepared latex particles for (a) PAM (b) PAM-Ia and (c) PAM-IIIa.

1680 cm^{-1} can be assigned to the composite of the ester groups of the surfmers and carboxamido groups of the acrylamide units [31]. All these bands from spectra (a), (b) and (c) confirm that the surfmers were successfully copolymerized with AM.

Also, the chemical structures of the HM-PAMs can be confirmed from their ^1H and ^{13}C NMR spectra. The ^1H NMR spectra of polyacrylamide (PAM), Poly(AM-co-OSA-eo22Ac) (PAM-Ia) and Poly(AM-co-O-OS-eo22Ac) (PAM-IIIa) are illustrated in Fig. 3(a–c). The ^1H NMR spectrum (3a) shows chemical shifts between δ (1) and (2.5) for ^1H protons (a) and (b) in $-\text{CH}_2$ and $-\text{CH}$ of acrylamide units. However, ^1H NMR spectrum (3b) shows chemical shifts at δ (0.7), (1) and (1.2) for ^1H protons (a), (b) and (c) in $-\text{CH}_3$ and $-\text{CH}_2$ of alkyl chain, a chemical shift at δ (3.5) for ^1H proton (d) in CH_2 of polyethylene oxide units. ^1H NMR spectrum (3c) shows the same chemical shifts as that in Fig. 3b in addition to a chemical shift at δ (3.9) for ^1H proton (e) in $-\text{CH}_2$ of the second alkyl chain that may be near the ester group. From all the above chemical shifts in Fig. 3a–c, it can be confirmed that the surfmers (of both types single or double tailed) were incorporated in the polymer backbone to produce the HM-PAMs. While, The ^{13}C NMR spectra of polyacrylamide (PAM), Poly(AM-co-OSA-eo22Ac) (PAM-Ia) and Poly(AM-co-O-OS-eo22Ac) (PAM-IIIa) are illustrated in Fig. 4(a–c). The ^{13}C NMR spectrum (4a) shows chemical shifts between (30) and (45) for ^{13}C carbons (a) and (b) in $-\text{CH}_2$ and $-\text{CH}$ of the acrylamide units, also the chemical shift at (180) for ^{13}C carbons (c) in carbonyl of the amide group of acrylamide units. The ^{13}C NMR spectrum (4b) shows chemical shifts between (10) and (30) for ^{13}C carbons (a) and (b) in $-\text{CH}_3$ and $-\text{CH}_2$ of alkyl chain and the chemical shift at (70) for ^{13}C carbon (c) in $-\text{CH}_2$ of the polyoxyethylene oxide units and finally a chemical shift

at (170) for ^{13}C carbon (d) in the carbonyl ester group. The ^{13}C NMR spectrum (4c) shows the same chemical shifts as that in Fig. 4b in addition to a chemical shift at (64) for ^{13}C carbon (c) in $-\text{CH}_2$ of the second alkyl chain which may be near the ester group. From all the above chemical shifts in Fig. 4a–c, it can be confirmed that the surfmers (of both types single or double tailed) were successfully incorporated in the backbone of the copolymers.

3.2. Thermal gravimetric analysis

The thermal stability of the prepared HM-PAMs was investigated by the thermal gravimetric analysis (TGA) for the polyacrylamides (PAM), Poly(AM-co-OSA-eo22Ac) (PAM-Ia) and Poly(AM-co-O-OS-eo22Ac) (PAM-IIIa) as shown in Fig. 5. The thermogravimetric curves of the copolymers showed three steps for the weight loss the same as that of the polyacrylamide homopolymer [32,33]. The first one occurred in the range of 20–200 °C (weight loss was about 13% of the total weight), corresponding to the evaporation of intra and intermolecular moisture. The copolymers contained hydrophilic groups which could induce the sample to combine with water molecules. The second one occurred in the range of 200–300 °C (weight loss was about 10% of the total weight), corresponding to the imine reaction of a small number of the amide groups and the thermal decomposition of hydrophobic side chains. The third one occurred in the range of 300–450 °C (weight loss was about 45% of the total weight) corresponding to the decomposition of amide groups and the degradation of the polymer main chains. Beyond about 450 °C, the copolymer decomposed completely. This part of weightlessness was due to the thermal decomposition of the

copolymer backbone [34,35]. From Fig. 5, the initial decomposition temperature (about 210 °C) for the copolymers is obviously lower than that (220 °C) of the polyacrylamide homopolymer [33], indicating that the incorporation of the alkyl chains of the surfmers into the polymer backbone minimize their thermal stabilities by a small magnitude.

3.3. Size of microemulsion droplets, latex particles and latex particles Morphologies

The size of the prepared microemulsion droplets and latex particles was measured by the DLS as shown in Fig. 6 for the polyacrylamide (PAM), Poly(AM-co-OSA-*eo*22Ac) (PAM-Ia) and Poly(AM-co-O-OS-*eo*22Ac) (PAM-IIIa) as representative samples. Fig. 6a shows that before polymerization the diameters of the prepared microemulsion droplets were 5, 15 and 17 nm with Z-averages 49, 827 and 506 nm for PAM, PAM-Ia and PAM-IIIa, respectively. After polymerization the diameters of the prepared latex particles became 8, 35 and 127 nm with Z-averages 32, 125 and 547 nm for PAM, PAM-Ia and PAM-IIIa, respectively as shown in Fig. 6b.

The prepared latex particle morphologies were determined by HRTEM as represented in Fig. 7(a-c). They are all almost spherical shape nanosize particles.

3.4. Molecular weights of the prepared copolymers

The molecular weights of the prepared copolymers were estimated by two different methods. The first one was the viscosity average molecular weight. Where, the prepared HM-PAMs have approximate intrinsic viscosities and hence, approximate weight average molecular weights, within the range from 282 to 336 ml/g and from 0.93×10^6 to 1.18×10^6 g/mol, respectively. The second method was the gel permeation chromatography. The GPC method results were closer to those of intrinsic viscosities, within the range from 0.89×10^6 to 1.58×10^6 g/mol, all the results are listed in Table 2.

The molecular weight of the prepared polyacrylamide homopolymer was 5.01×10^6 according to intrinsic viscosity $[\eta]$ 755 ml/g.

4. Conclusion

Nine high molecular weight hydrophobically modified polyacrylamide copolymers were successfully synthesized by inverse microemulsion copolymerization reactions using three groups of nonionic Polymerizable surfactants. Their chemical structures were characterized by different spectroscopic techniques. Their thermal stabilities were justified by TGA. The prepared nanolatexes were estimated using DLS. Finally their molecular weights were measured by GPC and calculated from their intrinsic viscosities, they were in the range from 0.89×10^6 to 1.58×10^6 g/mol. These polymers should be used in the application of enhanced oil recovery.

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