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Efficient organic solvent and oil sorbent co-polyesters: Poly-9-octadecenylacrylate/methacrylate with 1-hexene

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ABSTRACT

In this work, highly absorbent cross linked co-polymer gels of cis-9-octadecen-1-ol with acrylic/methacrylic acid and 1-hexene in different molar ratios were synthesized by thermal polymerization techniques and studied. The polymers were characterized by Fourier Transform-Infrared Spectroscopy, Thermogravimetric analysis and Scanning Electron Microscopy techniques. Their non-ionic nature was verified with the help of transport number measurement by Wagner polarization technique. The polymers were found to have very high swelling capacities in organic solvents like chloroform, tetrahydrofuran, ethanol, diethylether, thiophene, pyridine, and benzene. These gels show maximum swelling for chloroform which reaches up to 750%. The swelling capacities for different oils like kerosene, gasoline, engine oil, and silicon oil, are also favorable. The swelling kinetics confirmed that the swelling followed second order kinetics. The chloroform retention time was also checked which established that the gels can be recycled and reused again and again. An experiment showing removal of kerosene from kerosene/water mixture is also performed. This confirms that these gels can be used in removing organic contaminants and oil from water and can help in water purification and environmental cleanup purposes.

1. Introduction

Polymer gels are highly cross-linked networks [1] which have a wet and soft appearance [2] but are capable of absorbing large amounts of polar/nonpolar solvents from their environment. Depending on their chemical structure, these gels can absorb solvents up to several times their original dry weight [3]. Gels having good hydrophobicity, a fast solvent absorbing capacity [4] and reusability [5] were used as absorbents in different industrial applications. These gels were used as separating agents in different industries [6], as diapers [7], and also in medical applications such as drug delivery devices [8,9]. Organic solvent [10] and oil absorbing [11] polymer gels are also used for sorption of aromatic compounds from water [12], treatment of split oils [13] and thus help in environmental cleanup.

These gels due to their highly cross linked structures are insoluble in polar and nonpolar solvents [14] and have high thermal stability [15]. After swelling their physical properties change i.e. volume changes and they become softer than before. Most of these gels are either self cross linked polymers or they are polymerized in presence of small amount of crosslinker such as ethylene glycol dimethacrylate (EGDMA), and glutaraldehyde [16].

Absorbent materials find applications in collection and complete removal of the oil from oil spill sites and thus help in preventing environmental pollution. Organically modified clays [17], silica [18], exfoliated graphite [19], elastomers [20], cellulose-based materials [21] and carbon nanotubes [22,23] are extensively used as oil absorbents in several industries. These materials are porous and are able to absorb oil from water and thus can be used in water purification processes. Synthetic organic polymers such as polypropylene and polyurethane foams are the most commonly used oil absorbents due to their oleophilic and hydrophobic characteristics. Such materials provide proper disposal of oils and also check environmental pollution [24]. Few experiments have been carried out showing the recyclability of the absorbent materials and also showing efficient methods to recollect the absorbent oil from these substances [25].

Cis-9-octadecenyl polymer gels because of their large alkyl moiety absorb organic solvents and oil from the environment. In this work, we present co-polymer gels synthesized from cis-9-octadecenol and acrylic/methacrylic acids with 1-hexene, and the swelling properties are checked in different polar/nonpolar solvents and in different oils. All these polymer gels are found to have very high absorbing capacities in organic solvents as well as in different oils.

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2. Experimental

2.1. Materials

Cis-9-octadecen-1-ol (MERCK), methacrylic acid (SPECTROCHEM), p-toluene sulphonic acid (LOBA CHEMIE), ethylmethylketone (MERCK), 1-hexene (SPECTROCHEM), ethylene glycol dimethacrylate (MERCK) were used without further purification.

2.2. Synthesis of co-polymers with acrylic acid

About 0.03 mol (9.57 ml) of cis-9-octadecen-1-ol, 0.03 mol (2.5 ml) of acrylic acid, 0.05 g of p-toluenesulphonic acid were heated together for 30 min at 100–110 °C. After that 0.03 mol 1-hexene (1:1) was added to the reaction mixture along with 0.5 ml of ethylene glycol dimethacrylate (EGDMA, crosslinker) and allowed to react at 100–120 °C for 4 days. We have chosen thermal polymerization to avoid the use of any external agent. The above reaction was repeated with 0.06 mol 1-hexene (1:2) mol ratio. The products obtained were washed with ethylmethylketone and then vacuum dried to give 8 g and 8.2 g of soft yellow polymeric gels of poly-9-octadecenylacrylate-co-hexene. The co-polymers having 1:1 and 1:2 mole ratios of 9-octadecenylacrylate and 1-hexene were labeled as PODAH1 and PODAH 2 respectively.

2.3. Synthesis of co-polymers with methacrylic acid

0.03 mol (9.57 ml) of cis-9-octadecen-1-ol, 0.03 mol (3 ml) of methacrylic acid, 0.05 g of p-toluenesulphonic acid were heated together for 30 min at 100–110 °C. After that 0.03 mol 1-hexene along with 0.5 ml of EGDMA (cross linker) was added to the reaction mixture and allowed to react at 100–120 °C for 4 days. The same experiment was repeated with 0.06 mol 1-hexene (1:2) mol ratio. The products obtained were washed with ethylmethylketone and then vacuum dried to give 8.1 g and 8.2 g of soft yellow polymeric gels of poly-9-octadecenylmethacrylate-co-hexene. The co-o-polymers with 1:1 and 1:2 molar ratios were labeled as PODMAH1 and PODMAH 2 respectively.

2.4. Characterization

Fourier Transform Infrared Spectra of the monomers 9-octadecenylacrylate (ODA), 9-octadecenylmethacrylate (ODMA) and the co-polymers poly-9-octadecenylacrylate-co-hexene (PODAH1, PODAH2) and poly-9-octadecenylmethacrylate-co-hexene (PODMAH1, PODMAH2) were recorded with a Bruker Vector 22 FT-IR spectrophotometer in the range of 400–4000 cm^{-1}. The FT-IR spectra for the monomers were taken in the form of liquid. The polymer samples for FT-IR measurements were prepared in the form of pellets by mixing about 20 mg of IR spectroscopic grade potassium bromide with about 2 mg of dried samples. The spectra were recorded in transmission mode over 64 scans. The Thermogravimetric analysis (TGA) of the polymers was carried out using Perkin Elmer TGA 4000 at the heating rate 10 °C/min and with a nitrogen flow rate of 20 ml/min. The solubility of the polymers was checked in different polar and nonpolar solvents.

2.5. Morphological studies

Square shaped gels of size about 2 mm height were dried in a desiccator and considered for surface studies. The surface morphology of the gels was determined by SIGMA-VP (ZEISS) Scanning Electron Microscope at an accelerating voltage of 5 kV and at a constant magnification of 2000×. All the samples were gold coated before SEM analysis.

2.6. Transport number determination

The total ionic transport number, t_{ion} was evaluated by the standard Wagner polarization technique. The cell SS/(Polymer)/SS (SS stands for Stainless Steel) was polarized by a step potential (about 1 V) and the resulting potentiostatic current was monitored as a function of time. The two stainless steel plates act as blocking electrodes for the above cell. The t_{ion} was evaluated using the formula: $t_{ion} = (i_t - i_e)/i_t$, where i_t and i_e are total and residual current respectively.

In addition to the percentage electronic character of the polymers we also collected the XRD plots (Fig. S1) verifying amorphous nature of the gels and the change in conductivity of the gels (Fig. S2) with increase in temperature. These plots are given in the supplementary information.

2.7. Swelling measurements

The swelling measurement of the co-polymer gels was carried out in different polar/nonpolar solvents such as water, ethanol, acetone, tetrahydrofuran, diethylether, dichloromethane, chloroform, benzene, toluene, thiophene, furan, and pyridine, respectively, by gravimetric methods. For this purpose, a known amount of polymer sample was placed in a bag (made of filter paper) and immersed in a solvent. The weight of the filter paper bag was taken before placing the sample in it. The bags were taken out at different intervals of time (15 min, 30 min, 45 min, 60 min, 90 min and 120 min, respectively), blotted quickly to remove excess solvent, and weighed in a stoppered bottle. The swelling experiments were carried out at different temperature ranges i.e., 0 °C, 25 °C and 50 °C, respectively.

The swelling capacity can be calculated by the following formula:

$$\text{Swelling capacity} = \left[\frac{W_t - W}{W}\right] \times 100\% \quad (1)$$

where W_t is the weight of the swollen gel at time t and W is the weight of the dry sample.

The swelling capacities were also calculated for the gels in different oils, such as, kerosene, gasoline, engine oil (SAE 30), and silicon oil (DC 704). The known amount of dry gels were placed in different oils and left for 24 h. Then the gels were wiped with a blotting paper and their weights were recorded. An experiment showing the removal of kerosene by the co-polymer gel PODAH from water/kerosene mixture was also performed to prove their future applicability as water purifiers.

Temperature dependence of swelling in the above solvents and oils was measured at different temperatures i.e. 0 °C, 25 °C and 50 °C respectively. The sample was placed in the above solvents and kept at different temperatures until the swelling reached the equilibrium. The gels were taken out at different intervals of time and their weights were measured. Due to low boiling points, the swelling properties of the gels in solvents like tetrahydrofuran, chloroform, dichloromethane, and ether were not checked at 50 °C.

2.8. Kinetic studies

For kinetic studies, the solvent absorption capacities were measured by immersing a known amount of dry gel in polar/nonpolar solvents and then taking the weight of the swollen gel at different intervals of time (15 min, 30 min, 45 min, 1 h, etc.). Longer immersion times leads to greater absorption until the saturation point is reached. Thus we checked the swelling kinetics for all the gels [26]. For first order kinetics, the swelling is expressed as shown in the following equation:

$$\frac{dW_t}{dt} = K(W_\alpha - W_t) \tag{2}$$

where W_t is the weight of the swollen gel at time t, W_α is the weight of the swollen gel at equilibrium.

From Eq. (2), we get, $\ln W_\alpha/(W_\alpha - W_t) = Kt$.

If $\ln W_\alpha/(W_\alpha - W_t)$ vs. t gives a straight line, the swelling process follows a first order kinetics.

The expression for the second order rate for the swelling process is shown in the following equation:

$$\frac{dW_t}{dt} = K(W_\alpha - W_t)^2 \tag{3}$$

Integrating:

$$\frac{t}{W_t} = \frac{1}{KW_\alpha^2} - \frac{1}{W_\alpha}t \tag{4}$$

If the plot of t/W_t vs. t gives a straight line, the swelling tends to follow second order kinetics. The swelling rate can be calculated from the following equation:

$$S_t = S_e(1 - e^{-t/\tau}) \tag{5}$$

where S_t is the swelling at time t, S_e is the equilibrium swelling (power parameter) and τ is the rate parameter. The τ value is a measure of swelling rate (i.e. the lower the τ value, the higher is the rate of swelling).

3. Results and discussions

The reactions between cis-9-octadecen-1-ol and acrylic/methacrylic acids give their respective ester monomers. The monomers when heated with 1-hexene (different mole ratios) at 110–120 °C for 3–4 days in presence of a crosslinker EGDMA we get the copolymers PODAH1, PODAH2, PODMAH1 and PODMAH2. These reactions are summarized in Fig. 1a. All the polymers are highly insoluble in both polar and nonpolar solvents such as water, ethanol, tetrahydrofuran, benzene, chloroform, and dichloromethane, but the compounds have a very high swelling capacity in most of these solvents.

3.1. Fourier Transform-Infrared studies

Structural evidence of the polymers obtained from FT-IR spectroscopy is shown in Fig. 1b. The IR spectra of the monomer ODA and its copolymer PODAH 1 are compared. In ODA, the peaks at 2934 cm^{-1} and 2847 cm^{-1} are due to sp^2 and sp^3 C—H stretch, respectively. The peak at 1720 cm^{-1} is due to C═O stretch and that

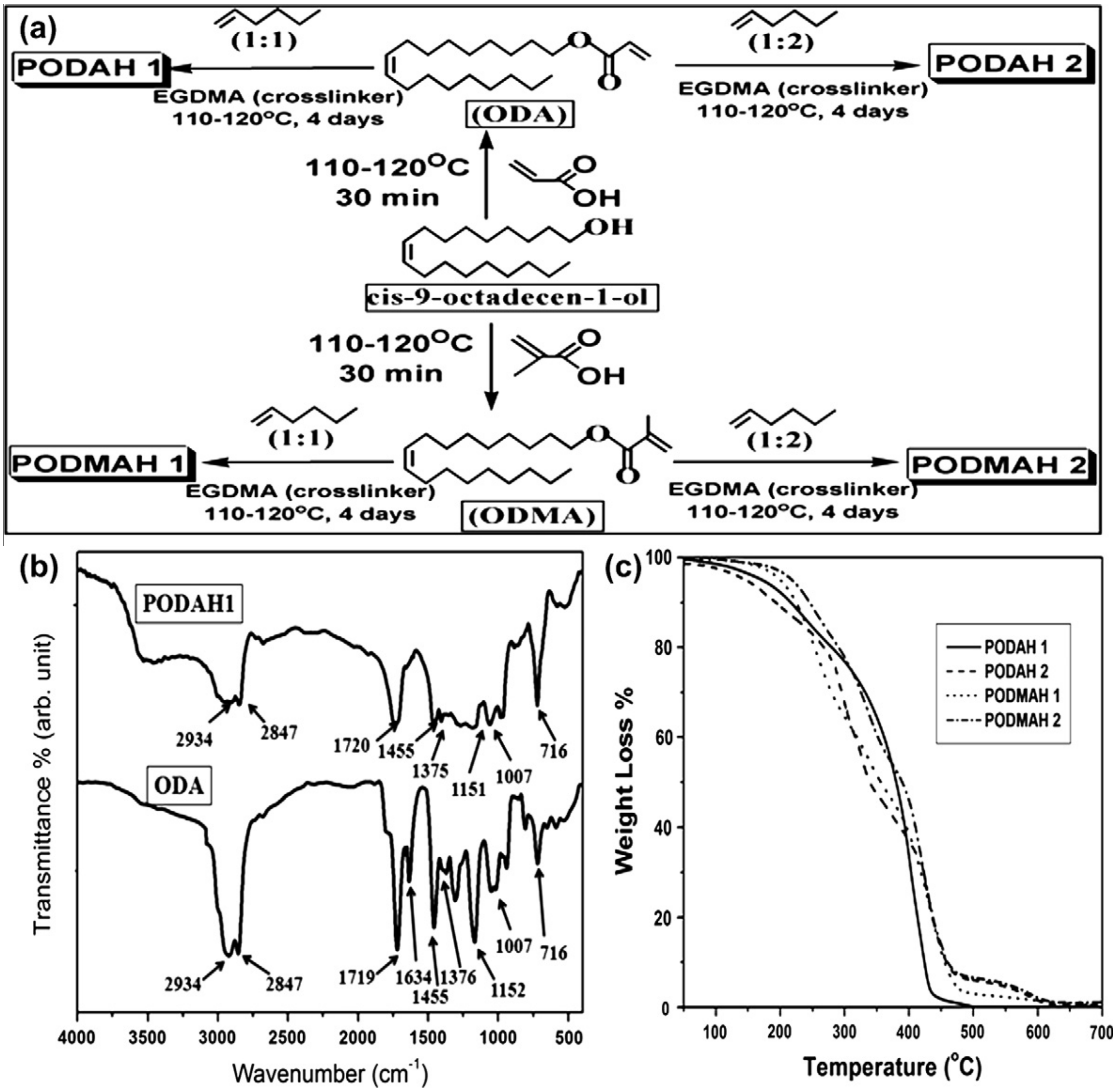

Fig. 1. (a) Schematic representation of the reactions involved in the process, (b) FT-IR spectra of ODMA, PODMAH1, (c) TGA plots of the gels.

at 1007 and 1151 cm^{-1} are due to C—O stretch of the ester group. The peaks at 1635 cm^{-1} and at 1654 cm^{-1} are due to C═C stretch of the terminal double bond and of the double bond at 9th position of the alkene group. The peaks at 1455 cm^{-1} and that at 1376 cm^{-1} are due to —CH$_2$ bend and —CH$_3$ bend. The peak at 716 cm^{-1} represents long chain band for the compound. In PODAH 1, the disappearance of the peak at 1635 cm^{-1} (due to the terminal C═C bond) indicates complete polymerization of ODA at the terminal end. In

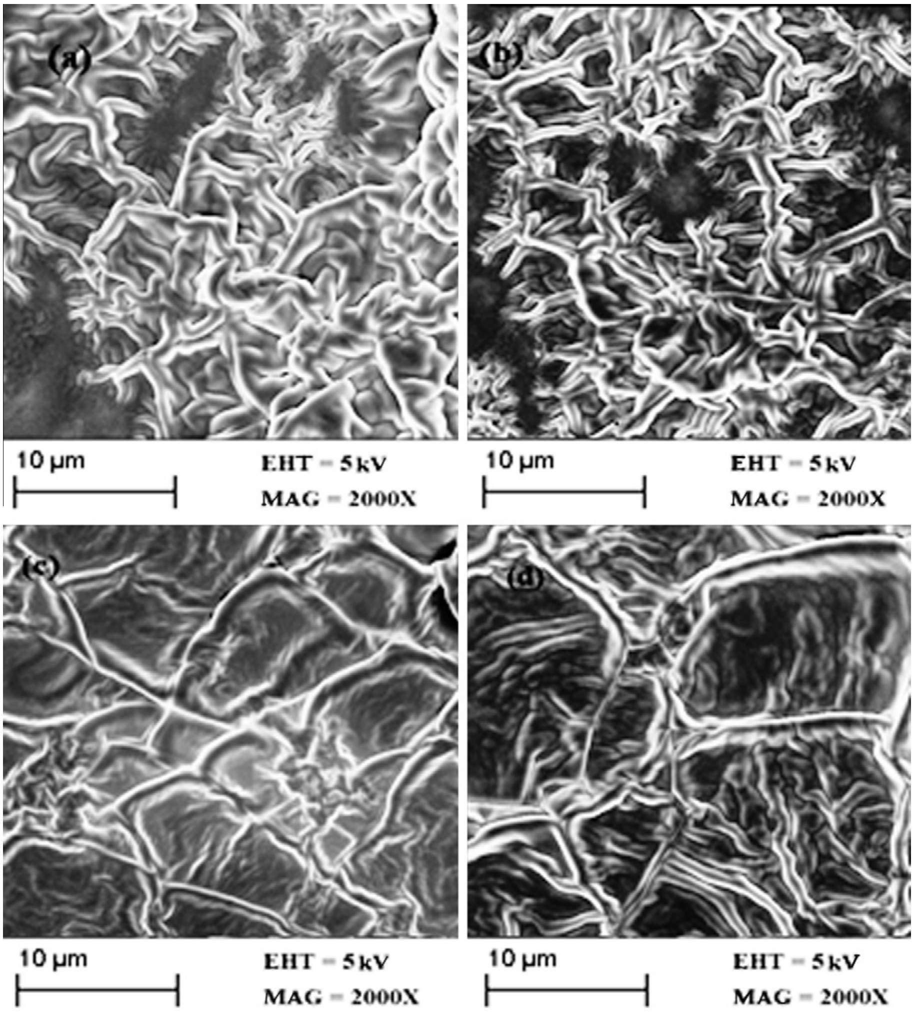

Fig. 2. Scanning Electron Micrographs of the gels (a) PODAH1, (b) PODAH2, (c) PODMAH1 and (d) PODMAH2.

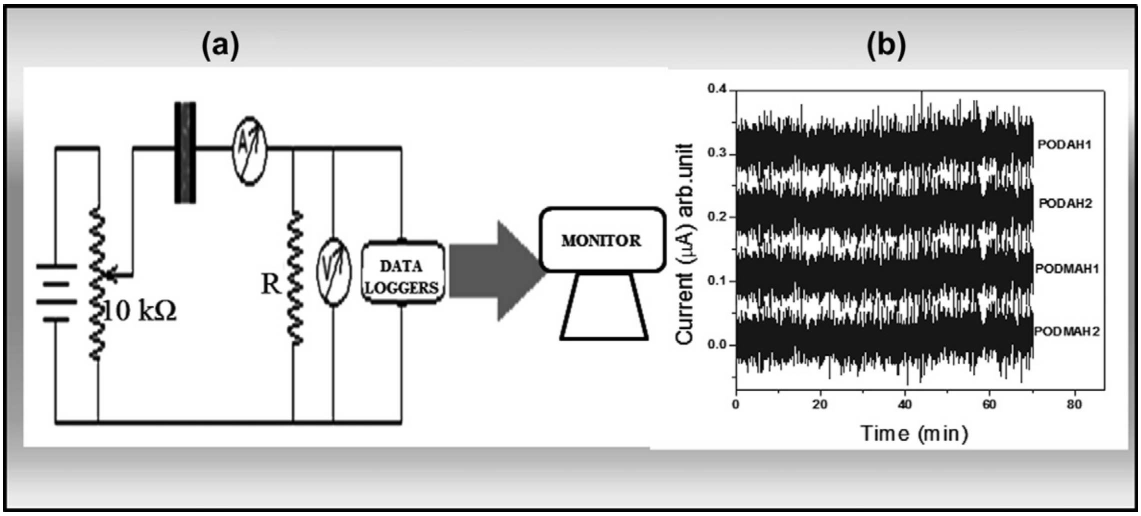

Fig. 3. (a) Schematic representation of cell circuit used to determine transport number, (b) current vs. time plot of the gels.

PODAH 1 a very sharp intense peak is observed at 716 cm^{-1} due to the long chain band in the copolymer.

3.2. Thermogravimetric analysis

The thermal stability of all the gels as shown in Fig. 1c were done in the temperature range 50–700 °C. The gels are thermally stable up to about 200 °C with a weight loss of 8% for PODAH1, 9% for PODAH2, 5% for PODMAH1 and 3% for PODMAH2 respectively. Complete degradation of the co-polymers PODAH1 and PODAH2 occurs at about 500 °C and 550 °C while that of PODMAH1 and PODMAH2 takes place at a higher temperature i.e. 615 °C and 650 °C respectively.

3.3. Morphological studies

The SEM images of the gels were taken at a constant magnification 2000× with an applied potential of 5 kV. The pictures in Fig. 2 shows that the gels have a highly porous network structure and the morphology of the co-polymers are almost similar. The pore sizes are larger in the case of the gels PODMAH than that of PODAH. Hence the gels PODMAH1 and PODMAH2 are better absorbents for most of the organic solvents and oils than the gels PODAH1 and PODAH2.

Table 1
Swelling capacity of the gels at 25 °C.

Solvents	Swelling capacity (%)			
	PODAH1	PODAH2	PODMAH1	PODMAH2
Chloroform	480	530	690	750
THF	425	456	510	537
DCM	384	402	431	455
Diethylether	320	331	385	397
Thiophene	278	292	330	345
Benzene	182	185	210	235
Toluene	220	234	245	269
Pyridine	115	131	186	213
Furan	145	158	167	185
Kerosene	135	158	204	220
Gasolene	147	163	225	237

3.4. Transport number determination by Wagner polarization method

The transport number of the polymers was determined employing Wagner polarization technique. Accordingly, on application of voltage to the cell, ionic migration occurs until steady state is reached. At the steady state, the cell is polarized and any residual current flows because of electron migration across the sample and interfaces. For ionic samples, the currents passing through an ion-blocking electrode falls rapidly with time. A cell is prepared by keeping the sample (in the form of thin film) in between two stainless steel envils. A step potential of about 1 V is then applied and resulting potentiostatic current is monitored as a function of time. The stainless steel envils act as ion blocking electrodes for the above cell. The circuit diagram of the experimental setup and the cell is shown in Fig. 3a.

For the experimental polymers the current vs. time plot, Fig. 3b shows no initial decrease in the total current with respect to time as is mostly seen in case of ionic conduction. This indicates that all the co-polymer gels are nonpolar in character devoid of any ions and therefore the gels are totally neutral in nature.

3.5. Organic solvent absorption

The solvent absorption capacities were measured by taking a known amount of the polymer in the organic solvent and then measuring the weight of the polymer at different intervals of time (15 min, 30 min, 45 min, 1 h, etc.). The saturation time was found to be about 90 min for all the polymers. The swelling capacities were checked at different temperatures (0 °C, 25 °C, 50 °C). The highest swelling capacities of the gels in most of the solvents at room temperature (25 °C) are given in Table 1.

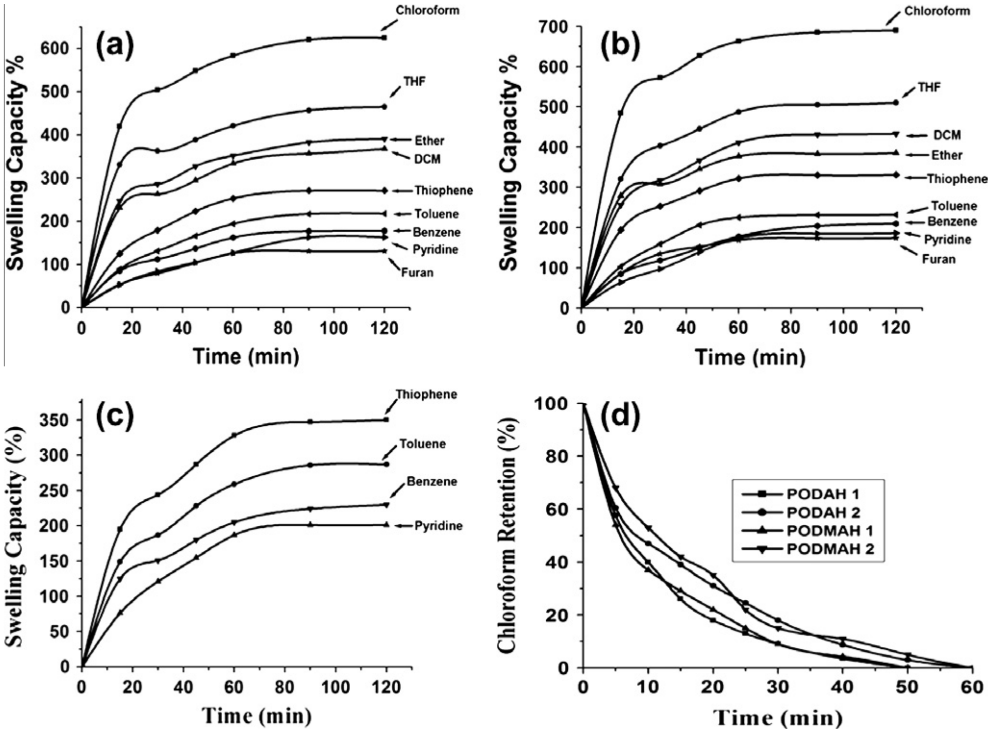

Fig. 4. Swelling capacities of PODMAH1 at, (a) 0 °C, (b) 25 °C, and (c) 50 °C, (d) chloroform retention of the gels in air.

All the polymers have very high swelling properties in organic solvents like tetrahydrofuran (THF), chloroform, ether, dichloromethane (DCM), ethanol, and toluene, in heterocyclic solvents like furan, thiophene and in highly carcinogenic solvents like pyridine, and benzene. The swelling properties of all the gel PODMAH 1 at different temperatures is shown in Fig. 4a–c. It was clearly observed that with the increase in temperature swelling capacities of the gel increases.

As for the swelling capacities, it was found that the swelling reached highest in case of chloroform which reached 690% for PODMAH1 and 750% for PODMAH2 at 25 °C. The swelling is larger in case of methacrylate compounds because of the presence of methyl group. The gels were found to have greater swelling properties in nonpolar solvents like benzene, chloroform and also in different oils because of the presence of large alkyl groups. Due to the same reason these gels do not swell in polar solvents like ethanol and water.

3.6. Recyclability of the swollen gels

The retention time for all the swollen gels was checked in chloroform by calculating the weight loss of swollen polymers at regular intervals of time (i.e. 5 min, 10 min, 15 min, etc.) as shown in Fig. 4d. All these experiments were carried out at room temperature. The swollen gels released the absorbed solvents to get back to their original size in about 1 h. To check the recyclability, we immerse these dry gels again in organic solvents. It was observed that the solvent absorption capacities of these gels did not change and the weight of the dry gel remained the same when used several times. This shows that the polymers are generable from most of the organic solvents and confirms its reusability in removing organic solvents and oil from the environment.

3.7. Hydrophobicity of the co-polymer gels

The hydrophobic nature of the gels was tested by dipping the gels in water at 25 °C and 50 °C for about 7 days and their swollen weight is compared with their dry weight. Experimental observations clearly shows that the gels do not show any water absorption property i.e. these gels do not get wetted when they are immersed in water. This is because of the presence of large alkyl group present in 9-octadecenylacrylate/methacrylate and the addition of nonpolar 1-hexene group which makes the gels highly hydrophobic. The co-polymer gels also do not swell in alcohols as was experimentally observed under identical conditions. This indicates that these gels may be used in water purification methods i.e. for removal of oil and other hazardous chemicals from water.

3.8. Oil absorption

The solvent absorption capacities were also checked in different oils like kerosene, gasoline, engine oil (SAE 30) and silicon oil (DC 704). The swelling was highest in gasoline and kerosene which reached 225% and 204% for PODMAH1 and 237% and 220% for PODMAH2 at room temperature. The swelling capacity is checked at different temperatures (0 °C, 25 °C and 50 °C respectively) and it is observed that swelling increases at higher temperatures. Thus the polymers are also useful in removing oil from the environment as is shown in Fig. 5.

Fig. 6a shows the photographs of the swollen PODMAH1 gels in chloroform, diethylether, tetrahydrofuran, and in gasoline. Pieces of the dry polymer gel of weight 0.1 g was immersed in the given solvents and the swollen gels were taken out. The photographs clearly show the efficiency of the gel as organic solvent and oil absorbents.

The co-polymeric gels can be of important industrial application in cleaning oil spills because of their water-repelling and efficient oil-absorbing properties. Fig. 6b illustrates the process of kerosene oil absorption from water/kerosene mixture by the co-polymer PODAH1 gel. A piece of PODAH1 gel of known weight was placed in a mixture of kerosene and water and kept undisturbed for two to 3 h. The gel floats on the oil surface and do not get dipped in water. It was observed that all of the oils on the water surface were quickly absorbed by the gel leaving behind pure water. This allows the

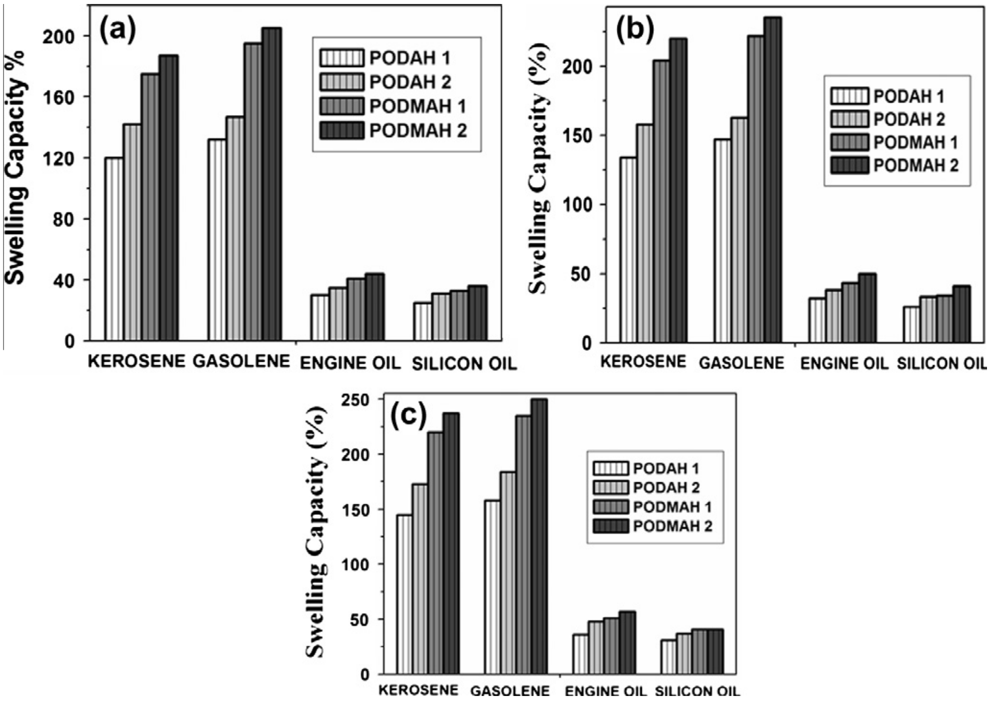

Fig. 5. Swelling capacities of the gels in oil at, (a) 0 °C, (b) 25 °C, and (c) 50 °C.

removal of kerosene oil from drinking water which is one of the major problems nowadays.

3.9. Swelling kinetics

A higher swelling rate is important together with a higher swelling capacity for practical applications. The study of absorption kinetics is significant as it provides valuable information about the reaction pathways and mechanism of absorption. The kinetic study describes the solvent uptake rate and helps in understanding the absorption process and its applications in water treatment (removing oil and other organic solvents from water) or environmental cleanup processes.

The plot of $\ln W_\alpha/(W_\alpha - W_t)$ vs. t for PODMAH1 swelling at room temperature is shown in Fig. 7a. All these measurements were carried out and it is found that the swelling of these gels in organic solvents do not follow first order kinetics. The plot of t/W_t vs. t from Eq. (4) is a straight line and it confirms that the swelling follows second order kinetics. These plots for the gel PODMAH1 at room temperature is shown in Fig. 7b.

The τ values for swelling of all the gels in chloroform, tetrahydrofuran, diethylether and kerosene at 25 °C are calculated and given in Table 2.

From the above τ values, it can be concluded that the swelling rate of the co-polymers are almost the same; the swelling of PODMAH1 and PODMAH2 being slightly faster than PODAH1 and PODAH2.

3.10. Effect of structure on swelling capacity

Gels swell only in those solvents that strongly solvate the polymer chains, thus breaking the intermolecular chains and replacing them with polymer–solvent interactions [27]. Neutral polymers when come in contact with organic solvents, the entropy

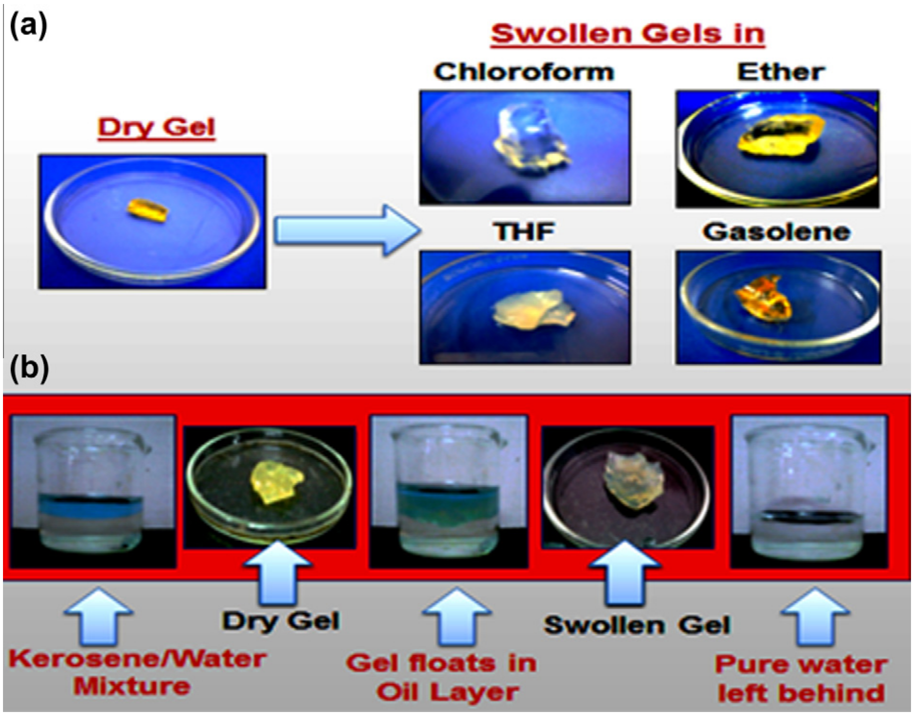

Fig. 6. (a) Photographs of PODMAH1 gel of equal weight swollen in different solvents. (b) Removal of Kerosene from water/kerosene mixture by PODAH1 gel.

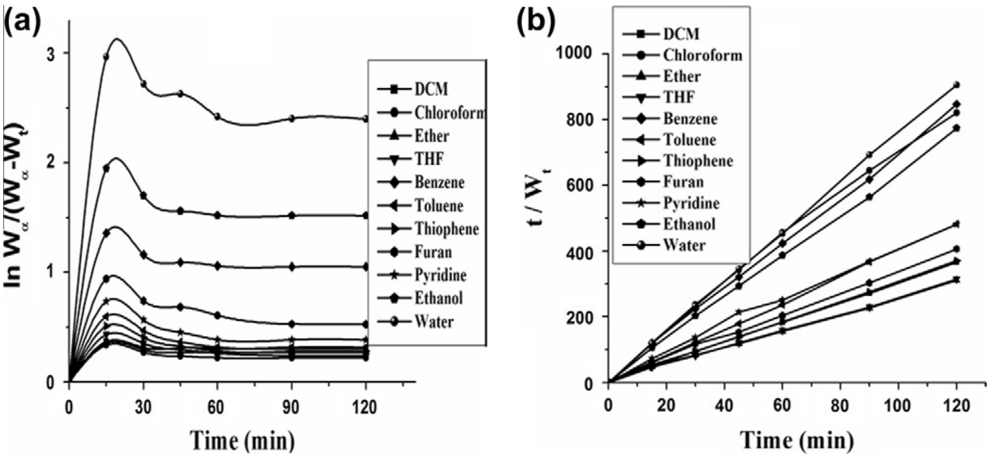

Fig. 7. (a) $\ln W_\alpha/W_\alpha - W_t$ vs. t plot, (b) t/W_t vs. t plot for PODMAH1.

Table 2
τ values of the polymers PODAH1, PODAH2, PODMAH1 and PODMAH2.

Solvents	τ (min)			
	PODAH1	PODAH2	PODMAH1	PODMAH2
Chloroform	15.8	16.8	15.3	16.2
Tetrahydrofuran	13.4	13.8	12.9	12.1
Kerosene	18.1	18.9	17.9	18.4
Diethylether	16.4	16.8	15.3	15.9

associated with it increases which causes stretching in the cross linked polymer chains resulting in swelling. The ODA and ODMA monomer units contain a large C-18 hydrophobic group together with the acrylate/methacrylate residues. The monomers when heated with 1-hexene (different molar ratios) in presence of the crosslinker EGDMA give their respective co-polymers. These co-polymers have larger pore sizes as observed from SEM studies. These are all highly cross linked neutral polymeric gels which are insoluble in most polar/nonpolar solvents. Again because of the presence of the large nonpolar hydrocarbon chain and addition of 1-hexene these gels are very good absorbents of organic solvents and different oils. They are strongly hydrophobic and do not show any swelling in highly polar solvents like water and ethanol. Conversely, due to their increased hydrophobic nature, they are better absorbents of oil and other organic solvents.

4. Conclusion

In this study, we demonstrated the synthesis and characterization of highly hydrophobic, nonionic co-polymeric gels of poly-9-octadecenylacrylate/(methacrylate)-co-hexene. The gels are found to be very good absorbents of organic solvents like chloroform, THF, ether, benzene, and pyridine. Maximum absorption is found in chloroform with a maximum of about 750%. The compounds also show high absorption capacities in kerosene and gasoline which crosses 200% in case of the methacrylate gels i.e. PODMAH1 and PODMAH2. Such gels can be of high practical use in industrial fields such as for removal of pollutants and other environmental cleanup purposes. Since the gels are recyclable they can be reused again and again for solvent absorption. The high hydrophobicity of these gels allows their applications in absorbing organic solvents and other oils from water and thus can be used as water purifiers. The experiment showing removal of kerosene from water/kerosene mixture establishes the above fact.

This study showed the synthesis of novel co-polymeric gels which can be used in organic solvent and oil absorption techniques. Further study on the effect of different cross-linking agents and reaction conditions in improving the absorption capacities of the compounds is under investigation.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.reactfunctpolym.2012.11.017.

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