



Synthesis, Characterization, and study the Performance for Oil/Water Separation using Cross linked Hydrophilic Copolymer-Coated Mesh

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Abstract:

Oil/water separation is an important issue that many industries have to address. In this work, hydrogels of ethylmethacrylate/mathacrylamide EMA/MAM copolymer with different compositions have been prepared using free radical polymerization technique. The polymerization was performed in the presence and absence of the crosslinking agent, methylene bisacrylamide (MBA). Metal meshes having different pore sizes (500 and 1000 micron) were then fabricated with the prepared hydrogels. Several parameters and properties have been studied to evaluate the performance of the prepared coated meshes as oil/water separator, water recovery, reclaimed oil, separation time, hydrogels coated percentages, swelling parameters, mechanical properties, microscopic and photographic images, and scanning electron microscopy. All the studied properties and diameters were directly affected by the used crosslinker amount, the composition of copolymer, and the pore size of coated meshes. From the experimental results water recovery in the range of 73 to 88 % was achieved. Separation efficiency increases as the amount of MBA increases in the hydrogels. The coated meshes having 1000 micron pore size show better separation efficiency than coated meshes having 500 micron pore size. Finally, copolymers with high MAM composition show excellent performance for oil/water separation. No study has been reported on preparation composite of EMA/MAM copolymer hydrogel-coated mesh as oil/water separator.

Keywords: Copolymer hydrogel; coated metal mesh; oil/water separation; mechanical properties

1. Introduction

It is estimated that 100 billion barrels of wastewater per year are produced by the oil and gas industry around the world (1). Therefore it can be seen that with the growing need to process oil and water mixtures, new innovative techniques to separate oil from water are desired. Moreover, one of the worldwide challenges is oil/water separation because of increase in the oil spill and industrial wastewater (2). Polymers have been widely used as additives or modifier materials to control the pollution in different situations (3). The description "hydrogel" refers to 3D network structures made of organic and/or natural polymers that can absorb and hold a large amount of water (4). The presence of polar hydrophilic moieties, such as SO_3H , OH , NH_2 , COOH , CONH_2 , as branched groups along the polymer network allows for the demonstration of such phenomena in hydrogels. The swelling parameters in water, mechanical and thermal properties, and many other properties are also affected by the degree of crosslinking in the polymer network (5). There are several methods in the literature have been adopted to separate oil from water such as induced gas floatation, centrifuge, media filter, API gravity

separator, hydro-cyclone, corrugated plate separator, mesh coalesce, and membrane filter (6-10). Hydrogel are compared to common existing products and methods to consider its feasibility for separation oil from water by membrane filter method. The most notable benefit with poly acrylamide derivatives membrane filter is that the chances of the hydrogel being fouled by the oil is minimum and there is no oil absorption is observed unlike media filter, and is evident from the hydro-philicity and underwater hydrophobicity of the hydrogel (11). Another benefit of hydrogels is that the procedure for synthesizing the hydrogel is simple process. The procedure of formulation with meshes is also relatively simple (12). Hydrogel formulation for the purposes of this project required only four chemical ingredients as well as deionized water and a metal mesh (13). The aims of this work are to synthesize crosslinked (ethyl methacrylate-co-methylacrylamide) hydrogels, to prepared composites composed of the prepared crosslinked polymers hydrogels and meshes with different pore sizes, and to study the performance of the prepared composites for oil/water mixtures separation.

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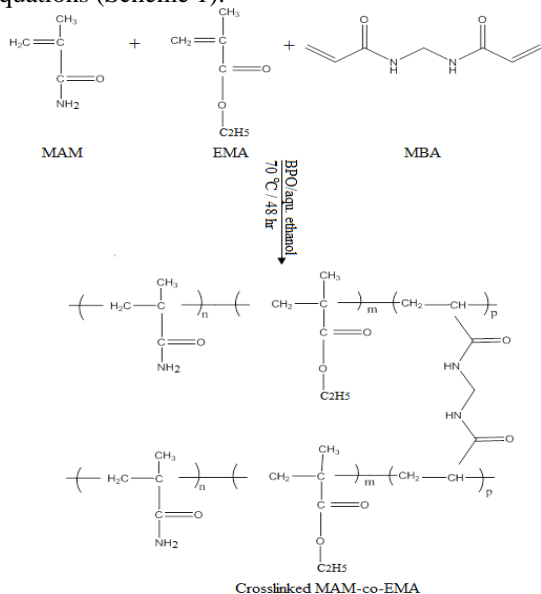
Experimental Section

Materials

Methacrylamide and methylene bisacrylamide (Aldrich-oma chemical Co) were first recrystallized from chloroform. Ethyl methacrylate (Merck chemical Co) was shaken 2-3 times with 10% NaOH to eliminate hydroquinone inhibitor, and then dried over anhydrous CaCl_2 for 10 h. Corn oil was collected from local market. Benzoyl peroxide (Aldrich-oma chemical Co) was recrystallized twice from methanol prior to use. All solvents were used as received.

Preparation of Hydrogels

Two systems of hydrogels were prepared, corresponding to the monomers and crosslinking agent concentrations. Monomers mixture was made up gravimetrically according to the desired composition (Table 1) in a small stopper flask. Mixture was magnetically stirred for 15 minutes before being moved to glass ampoules. Prior to the reaction, all oxygen was removed from the tubes by purging them with nitrogen for 15 minutes. To ensure complete conversion of the monomers to polymer forms, free radical initiated reactions were preferred at conventional temperatures, which ranged from about room temperature to about 100°C . In order to minimize the formation of bubbles in the polymers, polymerization was usually started at a temperature of $70\text{--}80^\circ\text{C}$. The glass ampoules were placed in a 70°C water bath and allowed to polymerize for two days. Then after, the temperature was increased, and the tubes were put in an oven at 90°C for another day. Polymerization usually completed at the end of this time (14). The reactions shown in the following equations (Scheme 1):



Scheme 1: Preparation reaction of crosslinked (MAM-co-EMA)

Table 1: Feed compositions of poly (MAM-co-EMA) hydrogels

Exp.	MAM% w/w	EMA% w/w	PBO% w/w	MBA %	Solvent vol ml (80% aq. ethanol)
1	50	50	0.30	0.0	15
2	80	20	0.30	0.0	15
3	50	50	0.30	3.0	15
4	80	20	0.30	3.0	15

Preparation of Mesh-Hydrogels Composites

After the copolymer solution has been homogenized, it is poured into a square dish to make coating the mesh pieces easier. As illustrated in Figure 1, the pre-cleaned mesh was carefully immersed in the solution for about 30 seconds before being slowly and horizontally taken out with the solution adhering to the mesh's surface. The mesh was dipped and then baked for one day at 80°C to thoroughly polymerize the solution. This enabled the coated solution to be thermally initiated and transformed into a polymerized hydrogel. To remove impurities left over from the preparation procedure, the coated mesh was washed with distilled water after polymerization. Unreacted monomers, cross-linkers, initiators, and other side reaction products are among them. The final hydrogel-coated mesh was thus prepared.

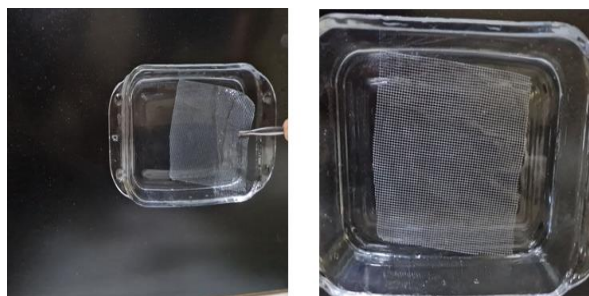


Figure 1: Preparation of mesh-hydrogels composites

Separation of Oil from Water Using Mesh-Hydrogels Composites

The hydrogel-coated mesh was sandwiched between two open plastic pieces (30 mm in diameter) that served as a separation membrane, and the oil-water mixture (v/v %) was then poured onto the upper glass tube on the hydrogel-coated mesh. In a beaker, 50 mL of 30% oil/70% water combination was made by mixing required amounts of the two components and stirring at 500 rpm for 25 minutes to form an emulsion, as illustrated in Figure 2. The oil remained in the upper of composite, whereas the water passed through the hydrogels. Separation time was also measured using digital clock.

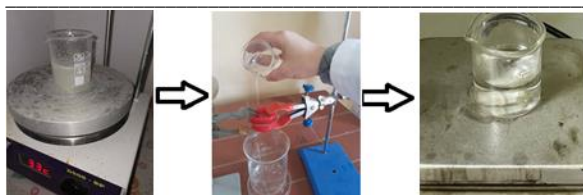


Figure 2: Separation process of oil/water mixture using coated mesh

Characterization

Perkin Elmer-1650 spectrophotometer completed with data processing facilities was used to determine the functional groups in the copolymers using a KBr disk method at wavenumber range of 400 to 4000 cm^{-1} . Shimadzu-3600 UV spectrophotometer was used to estimate the concentration of the oil after filtration and the separation efficiency according to a standard procedure.

Water Recovery and Reclaimed Oil

Water recovery and recaptured oil were assessed after separation. By employing equation 1, the water recovery is calculated as the proportion of water filtered through the mesh compared to the original water content in the testing mixture. $(V_{\text{water}})_{\text{filtrate}}$ is the volume of water in the filtrate, and $(V_{\text{water}})_{\text{initial}}$ is the volume of water in the initial feed mixture in equation 1. The reclaimed oil % is determined using equation 2, where $(V_{\text{oil}})_{\text{filtrate}}$ is the volume of the oil contained in the filtrate and $(V_{\text{oil}})_{\text{initial}}$ is the volume of the initial oil in the sample.

$$\text{Water Recovery} = \frac{(V_{\text{water}})_{\text{filtrate}}}{(V_{\text{water}})_{\text{initial}}} 100 \quad (1)$$

$$\text{Reclaimed Oil} = 1 - \frac{(V_{\text{oil}})_{\text{filtrate}}}{(V_{\text{oil}})_{\text{initial}}} 100 \quad (2)$$

Duration Time of Separation Process

Separation time was measured directly during the experiment and did not require any additional calculations. The separation time was measured from the moment the mixture was put onto the top of the coated mesh until no fluid droplets could be seen dripping from the mesh's bottom for at least three seconds.

Calculation of Coating Percentage with Hydrogel

The coating percentage with hydrogel was examined by using equation 3 (15).

$$\text{CPH} (\%) = \frac{(W_a - W_b)}{W_b} 100 \quad (3)$$

Where W_a is the mass of the coated mesh after polymerization and W_b is the mass of the uncoated mesh before immersion in the hydrogel. Based on varied mesh pore diameters (500 and 1000 micron), the percentage of hydrogel-coated mesh was calculated.

Estimation of the Residue Oil-in-Water

The residue oil-in-water after must be analyzed after separation. The oil was first extracted from water by

using suitable organic solvent (hexane). The organic layer is separated from the aqueous layer using a separator funnel. After separation, the oil content was calculated using the oil calibration curves in the hexane. The concentration of the oil after filtering was evaluated using a UV-VIS spectrophotometer in the separation system, according to a standard method.

Swelling

The gels were dried at room temperature for several days before being placed in a vacuum oven at 50°C until they reached a constant weight. Weighted xerogels discs were equilibrated in deionized water for several weeks before they reached equilibrium swelling. From the time the solvent was added, the swelling time was measured. The weight increment of the discs as a function of time was used to record swelling gravimetrically. The swollen piece of hydrogel was regularly removed from the water, gently placed between two pieces of filter paper to remove excess water from the surface, and then weighed, before being returned. The equilibrium water content (EWC) of a hydrogel was calculated using the weight of discs after (30 days completely hydrated) and the weight of the same discs after being dried in an oven at 50 °C for (48 hr). EWC was calculated as follows for the same sample (16):

$$\text{EWC} \% = \frac{(W_s - W_d)}{W_s} 100 \quad (4)$$

where W_s and W_d are the weights of the swollen sample after 30 days of complete hydration and after 48 hours of drying in a 50°C oven. The following relation was used to calculate the weight reduction of the xerogels:

$$\text{Weight loss during swelling} = \frac{(W_0 - W_d)}{W_0} 100 \quad (5)$$

where W_0 and W_d are the weights of the xerogel sample and the dried sample after 48 hours in a 40°C oven.

Scanning Electron Microscopy (SEM)

The surface topography of the sample was investigated by SEM using MIRA III apparatus in university of Baghdad. An acceleration voltage of 5 to 10 kV was used to capture the images. The coated mesh (0.5 cm) test specimens were fixed on aluminum stubs with carbon tape. Samples were sputter coated with 10 nm gold using a high-resolution sputter coater before the measurements began.

Light Microscopy

5 ml of oil was placed under light microscopy (EVOS fl digital inverted microscope) and 5 photographs were shot randomly from different regions using a camera microscope to determine the droplet size.

Tensile Testing

The tensile test was used to determine the mechanical properties of coated mesh. These tests were carried on an Instron 3366 machine in accordance with

ASTM 3039. The load was run at a constant speed of 2 mm/min until the sample was broken. The coated meshes were cut with dimensions of (100 mm in length and 9-10 mm in width).

Results and Discussion

The absorption bands which appear in the FTIR spectra of the copolymers belong to the stretching vibration in different functional groups of corresponding monomers. The structures of the copolymers EMA/MAM with different composition are shown in Figures 3. The absorption bands are as follows: the carbonyl absorption of EMA was observed at 1725 (ester C=O), 1650 (amide C=O) of MAM. A broad band at 3250 cm⁻¹ corresponded to the N-H asymmetrical stretching vibration of the amide group in MAM. A band at 1311-1384 cm⁻¹ corresponded to C-N and N-H stretching vibration of MAM. The sharp band at 1400-1450 cm⁻¹ is attributed to the C-N stretch vibration of MAM. The multiple bands at 1000-1250 cm⁻¹ belong to C-O-C in EMA. Furthermore, absence of olefinic double bond peak in the copolymers.

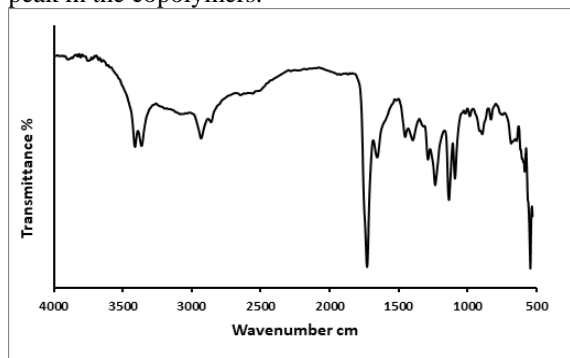


Figure 3: FTIR spectrum of EMA/MAM copolymer

Evaluation of Separation Process

Water Recovery

Figure 4 shows the water recovery percentage for different mesh sizes (500 and 1000 μm) based on two concentrations of MAM in the EMA/MAM copolymer (with and without crosslinker). It was observed that with an increase in the MAM concentration from 50 to 90 % in EMA/MAM copolymer, the water recovery increases as the hydrogel gets thicker. As the polymer concentration increases, viscosity of the solutions increases as well. On the other hand, the polymer with crosslinker is less water recovery than without crosslinker. Generally, a higher MBA content led to a stronger gel with lower water content. This was expected because of the increasing crosslinker density, which limited the diffusion of water molecules into the gel network. So that, the hydrogel with a MBA, absorbs lesser amount of water in the equilibrium swollen state. This finding has been frequently reported in many other investigations (17, 18). However, it can be seen

from Figure 4 that water recovery was ranging and fluctuating based on the mesh sizes and monomer concentrations. It is found that water recovery was ranged from 73 to 88 % for the two mesh sizes. Since the hydrogel holds some water the water recovery cannot go over 97 %.

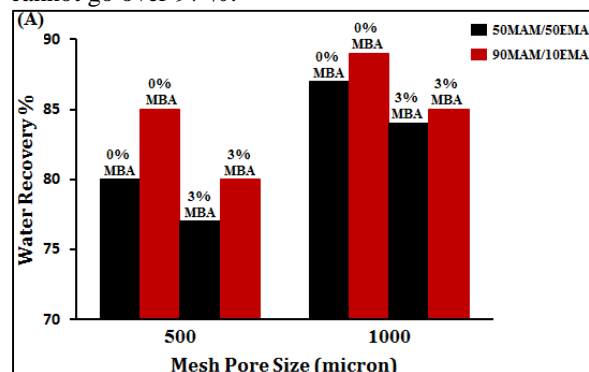


Figure 4: Water recovery of MAM/EMA copolymer with different composition

Residual Oil in Water

The probable presence of residual oil in the filtrated water was tested with UV-visible spectroscopy to confirm the excellent separation efficiency. Figure 5 shows the separation efficiency of MAM/EMA copolymer. The highest separation efficiency was for 10 % oil and 90MAM/10EMA/3%MBA in based on 1000 micron mesh was calculated as 95.3. Moreover, the separation efficiency has not been changed when the crosslinker was added.

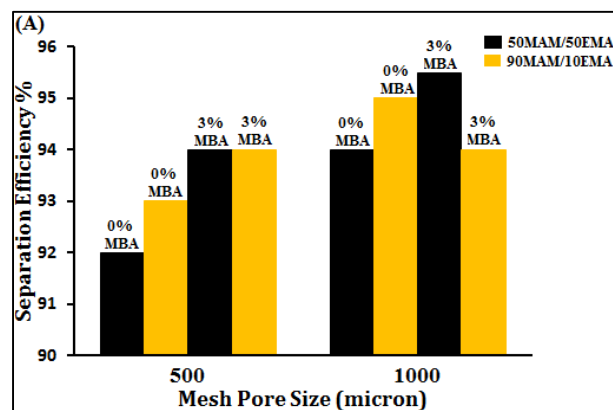


Figure 5: Separation efficiency of MAM/EMA copolymer with different composition

There is also a considerable difference in phase composition between the feed and the filtrate, as demonstrated by light microscopy images, as illustrated in Figure 6, where the collected filter (right) is transparent compared to the original mixture (left).

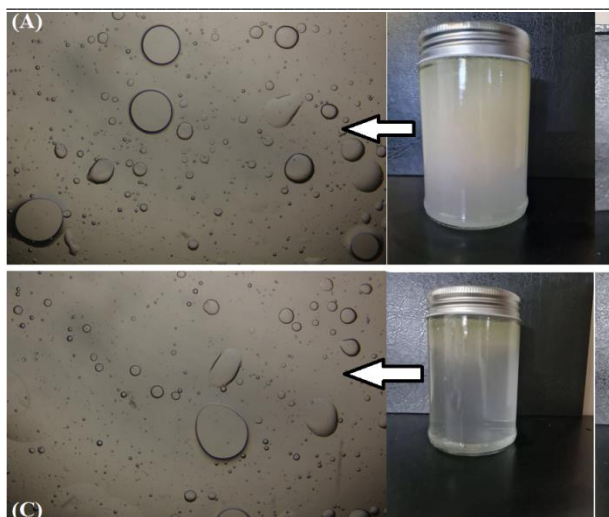


Figure 6: Microscopic and photographic images before (A and C) and after separation (B and D) based on the 1000 micron mesh

Duration Time of Separation Process

The separation time decrease as the mesh pores increased for 500 and 1000 micron (Figure 7). The copolymer with 50 % MAM monomer separated the fastest; this is because the hydrogel coating on the mesh is thinner with lower monomer concentration, resulting in faster separation. However, as the hydrogel became thicker, the separation time increased as the monomer content in the hydrogel increased. Thus, 90MAM/10EMA copolymer had more separation time than 50MAM/50EMA copolymer. In addition, it was observed that with increasing the crosslinker (MBA) amount in copolymer, results in increasing the separation time. This result was expected since the crosslinker decrease the swelling ability of hydrogels.

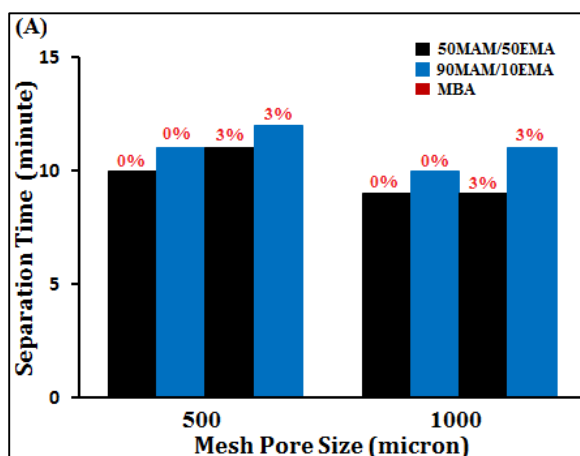


Figure 7: Separation time of MAM/EMA copolymer with different composition Coating Percentage with Hydrogel

Figure 8 shows that the smallest mesh pore size was coated with gel more than the coarse mesh, which increased the hydrogel's absorption ability and improved oil/water separation. Furthermore, raising

the hydrogel concentration increases the fraction of mesh that is hydrogel-coated. The hydrogel was thinner with low monomer content than with a greater monomer concentration. Thus, hydrogels composed of 90MAM/10EMA was thicker than hydrogels composed of 50MAM/50EMA, respectively. The separation time was faster, but the separation efficiency was lower than with a thicker hydrogel. Moreover, it can be clearly observed that as crosslinker concentration increase from 0 to 3%, the coating percentage increase. These results may be explained in term of increasing the crosslinking density of the hydrogels, so the hydrogel layer will be thicker. Hydrogel coating percentage measurements verified this result, which proved that hydrogel gets thicker with increasing total monomer and crosslinker concentration.

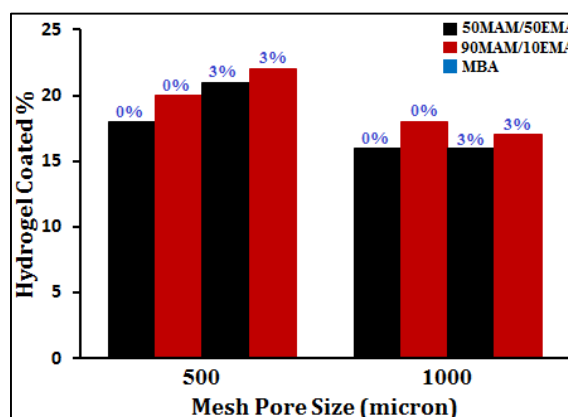


Figure 8: Hydrogel Coated % of 90MAM/10EMA and 50MAM/50EMA copolymers as a function of mesh sizes

Swelling Parameters

The EWC values of the hydrogels are shown in Figure 9, they are in the range of (90.01 – 53.86%). MAM50/EMA50 with MBA had the lowest swelling capacity while 90PMAM without MBA had the highest EWC. The absence of the crosslinker MBA allows an easy diffusion of water molecules outside of the gel phase. As expected, the amount of weight loss during swelling in the prepared hydrogels decreases with increasing MBA and EMA contents. At the beginning of the copolymerization the monomer composition is the same as that in the feed composition. But, since one of the monomers has a higher reactivity ratio, it will react more rapidly than the other, and the amount of that monomer will decrease in the feed more than the other comonomer. Therefore, the concentration of the monomer with the higher reactivity ratio will be almost zero before the end of the reaction. Thus, near the end of the reaction, the copolymer will contain mostly comonomer-2 because comonomer-1 has already fully reacted. Since EMA is more reactive than MAM, ($r_{EMA} = 0.203$ and $r_{MAM} = 0.232$) (19), it reaches the copolymer faster than MAM. As a result,

in the later stages of conversion, the substance has a structure that is very similar to EMA homopolymers, insoluble in water, and is extracted out on swelling, and has shown compatible effects with the weight loss results. The same behavior has been observed when the swelling property for *N,N*-dimethylacrylamide-co-maleic acid was studied (20). This is the common effect on the properties of polymers (20, 21).

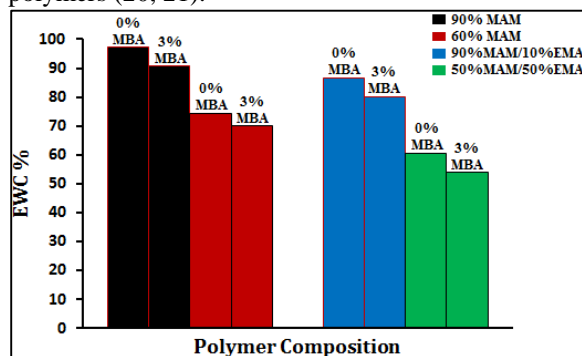


Figure 9: Equilibrium Water content (EWC) of PMAM and MAM/EMA copolymer with and without MBA

Mechanical Tests (Tensile Testing)

The results show that increasing MAM concentration leads to an increase in stress modulus. So that, the stress of 500 micron mesh coated with 90MAM/10EMA is more than 50MAM/50EMA. This behavior could be attributed to increase the monomer (MAM) concentration which led to increase the interaction with the mesh. On the other hand, when the crosslinker amount was 3% for 50MAM/50EMA, the stress was more than 50MAM/50EMA without crosslinker (Figures 10 and 11). The obtained results could be explained in term of increasing the crosslinker amount will restrict the mobility of polymer chain, thus the polymer will be stronger. This fact was discussed early by Mohammed et.al for (vinylpyrrolidone/(trimethoxysilyl) propyl methacrylate) system (15). The mechanical properties enhance as the pore size of mesh decrease from 1000 to 500 micron. These results could be attributed to the fact that interaction between 500 micron mesh and polymer is more than 1000 micron mesh.

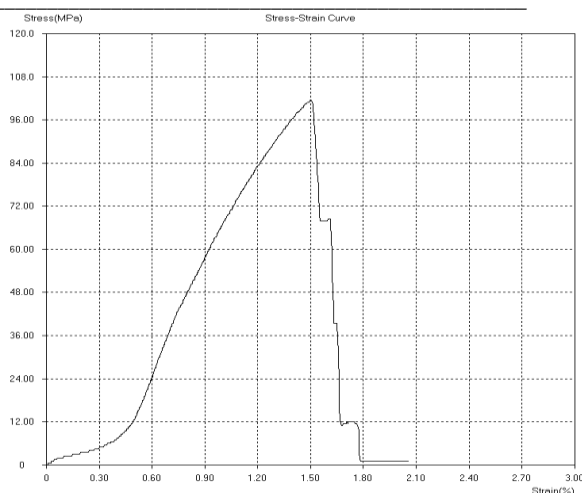


Figure 10: Tensile test of 500 micron mesh coated with 50MAM/50EMA

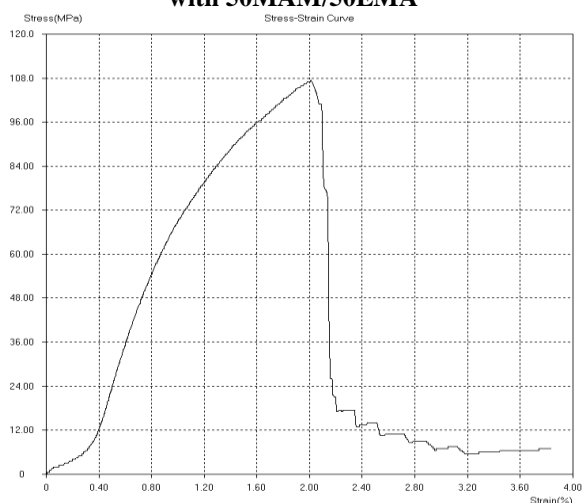


Figure 11: Tensile test of 500 micron mesh coated with 50MAM/50EMA/3% MBA

Scanning Electron Microscopy (SEM)

Scanning electron microscopy measurements were carried out to characterize the surface morphologies of the mesh before and after coating. Figures 12 (B and C) show the SEM images of MAM/EMA copolymer (50% MAM and 90% MAM) with 3% crosslinker coating mesh with a pore diameter of about 500 micron. Typical images of the uncoated mesh substrates with pores diameter of 500 microns is shown in (A), which indicates the original meshes have a smooth and clear surface. The results excited in this section are in agreement with the results of coating percentages, which indicate that the hydrogel was thinner with low monomer content than with a greater monomer concentration.

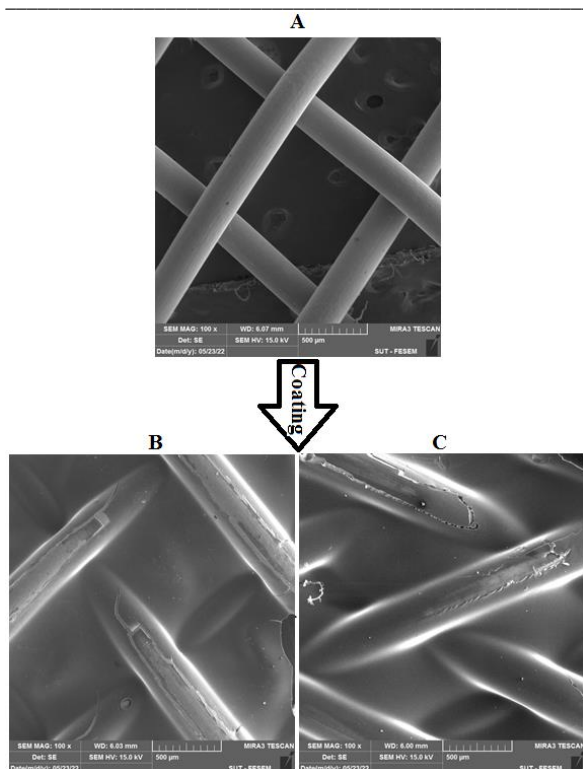


Figure 12: SEM images of A- uncoated mesh (500) microns, B- mesh coated with 50MAM/50EMA/3% MBA, C- mesh coated with 90MAM/10EMA/3% MBA

Conclusion

Reinforced hydrogels coated metal mesh was efficiently synthesized and used for oil water separations. 10EMA/90MAM and 50EMA/50MAM copolymers have been successfully prepared and tested for separation efficiency. The effect of the crosslinker amount, mesh pore size and the composition of the copolymer on the separation efficiency were evaluated. Hydrogel with 50EMA/50MAM composition formed thinner membrane than with 10EMA/90MAM composition. The separation time is faster with the thinner membrane but separation efficiency is less than that of thicker hydrogel. The separation time decreased with an increase in the mesh size from 500 to 1000 micron. The composition 90MAM/10EMA/3%MBA show a good performance when it is coated with meshes having 1000 micron pore size. Finally, the mechanical properties of all composites (mesh + hydrogels) were enhanced due to the presence of MBA as crosslinking agent.

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