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# Synthesis and characterization of isodecyl methacrylate monomer and its copolymer with acrylamide for oil/water separation

**Fatima Hekmat Assal and Ameen Hadi Mohammed**

Department of Chemistry, College of Science for Women, University of Baghdad



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**Corresponding Author**

E-mail: aminhm_chem@csw.uobaghdad.edu.iq

Mobile: 07739824122

**Abstract**

In this work, isodecyl methacrylate monomer IDMA was was synthesized by esterification of methacrylic acid with isodecylalcohol. IDMA monomer was then copolymerized with acrylamide monomer AAM, in different feed compositions, using free radical polymerization technique. The synthesized IDMA and IDMA/AAM copolymer were characterized by employing proton nuclear magnetic resonance and Fourier transform infrared spectroscopy techniques. Composites of fabricated polymers with metallic mesh, having different pore size, were prepared to examine their performance for separation oil from water. Separation time, reclaimed oil, coating percentage with polymer, and water recovery were directly affected by the composition of copolymer and mesh pore size. The highest separation efficiency of copolymer was for 25 % IDMA based on 1000 micron mesh which was calculated as 99%. Water recovery was ranged from 75% to 92%. 75AAM/25IDMA copolymer, without crosslinker, separated oil from water faster than other compositions. Microscopic and photographic images of water, before and after separation by polymers, showed that oil was completely removed from water.

## Introduction:

Acrylate monomers prepared from acrylic acid derivatives and alcohols have gained more interesting due to their importance in the synthesis of various polymers used in different industrial, agricultural, and biomedical applications [1, 2].

The term "hydrgel" is used to describe three-dimensional network topologies composed of natural or synthetic polymers capable of absorbing and retaining large amounts of water [3, 4]. The presence of Hydrophilic polar groups, such as $CONH_2$, COOH, $NH_2$, OH, and $SO_3H$ others, allows explaining this phenomenia in hydrogels [5, 6].

Separation of oil from water produced by medical and petroleum factories is considered as a serious problem that the researchers have to address [7, 8]. Many methods are used to minimize the effect of this problem, such as, Centrifuge, API gravity separator, Hydro-cyclone, Membrane filter, Induced gas floatation, corrugated plate separator, and Mesh coalesce [9, 10]. Polymers play an important role in synthesized superolophobic surfaces that can be used to separate oil from water, and they also have unique combination of properties, good biocompatibility and low cost [11, 12].

Many studies have discussed the use of polymers as oil/water separators. Arshad Hussain and Mohammed Al-Yaari developed Nylon 66 blended with cellulose acetate (CA) membranes blended to treat the oily wastewater; the results are compared with a commercial membrane of cellulose acetate. Li Gong and his coworkers developed new composite composed of SHMP-1@Sponge and skeleton of 3D melamine sponge to separate oils and other organic solvents spilled into water [14]. In another study, an oil/water separation unit was designed using a superhydrophobic poly(vinylidede) fluoride (PVDF) membrane with artificially adjustable surface topographies [15].

The main aims of this work are to synthesis and characterize isodecyl methacrylate monomer, to copolymerize it with acrylamide monomer. This study is also aimed to fabricate the prepared copolymer with metallic mesh for using as oil/water separators. No study, in the literature, has prepared this type of monomer and copolymers as well as using this copolymer for oil/water separation.

**Experimental part**:

**Materials**

Methacrylic acid and isodecyl alcohol, and all solvents were purchased from Aldrich-oma chemical Co. Acryl amide monomer and potassium persulfate PPS initiator were collected from Merck chemical company and purified by recrystallization from methanol.

**Characterization**

The functional groups in the synthesized IDMA and IDMA-co-AAM were characterized using a Perken Elmer-1650 spectrophotometer at 400 to 4000 $cm^{-1}$ wavenumber.

**Synthesis of IDMA monomer**

A direct esterification method was used to synthesize isodecyl methacrylate monomer IDMA. Equal moles of methacrylic acid and isodecylalcohol in addition to 1% of hydroquinone as polymerization inhibitor were dissolved in 75 ml of benzene. After the reaction was catalyzed by adding 0.5 wt% of paratoluenesulfonic acid, the mixture was refluxed at 65 $^{o}C$ for 12 h. In order to remove the unreacted methacrylic acid and catalyst, the product was washed many times with ethanol/water mixture while benzene was removed by using rotary evaporator. The reaction is shown in Figure 1.

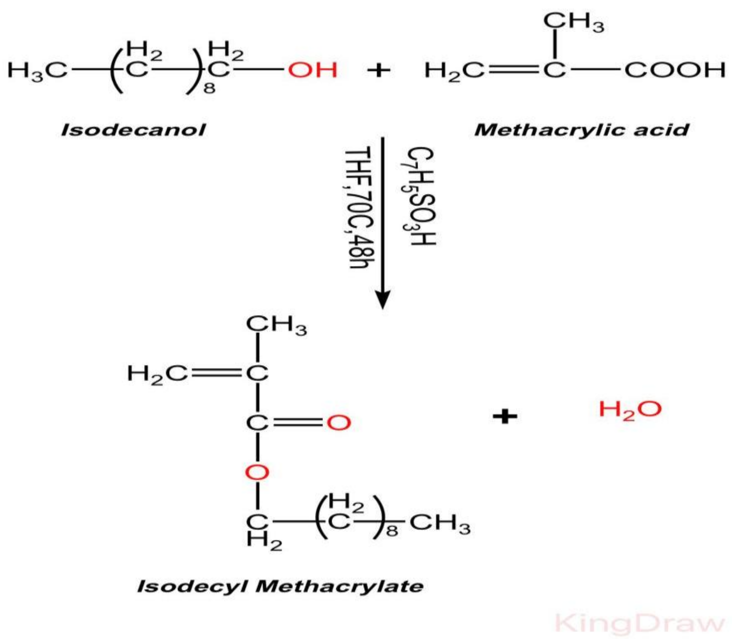


**Fig. 1** *Scheme of isodecyl methacrylate synthesis*

**Synthesis of IDMA/AAM copolymer and its composite with metallic mesh**

Different compositions of IDMA/AAM copolymers were prepared by changing the mole fractions of IDMA and AAM in the feed mixture to 25/75, 50/50 and 75/25. In quickfit glass test tubes, the desired amount of monomers and 0.001 gm of PPS as an initiator were dissolved in 25 ml of ethanol. The polymerization reaction was accomplished by placing the tubes in a thermostated water bath at 60 $^{o}$C for 48 h. To prepare the composite of polymer and metallic mesh, the polymerization solution was poured in a square plate, and the pre-cleaned mesh was submerged in the solution for one minute. To ensure completing the polymerization process and the strong adhesion of polymer with the mesh, the composite was put in a thermostat oven at 80 $^{o}$C for 24 h. Finally, the composites were washed many times with distilled water to remove the unreacted monomers and initiator. The polymerization reaction and fabrication of IDMA/AAM copolymer with metallic mesh are shown in Figures 2 and 3, respectively.

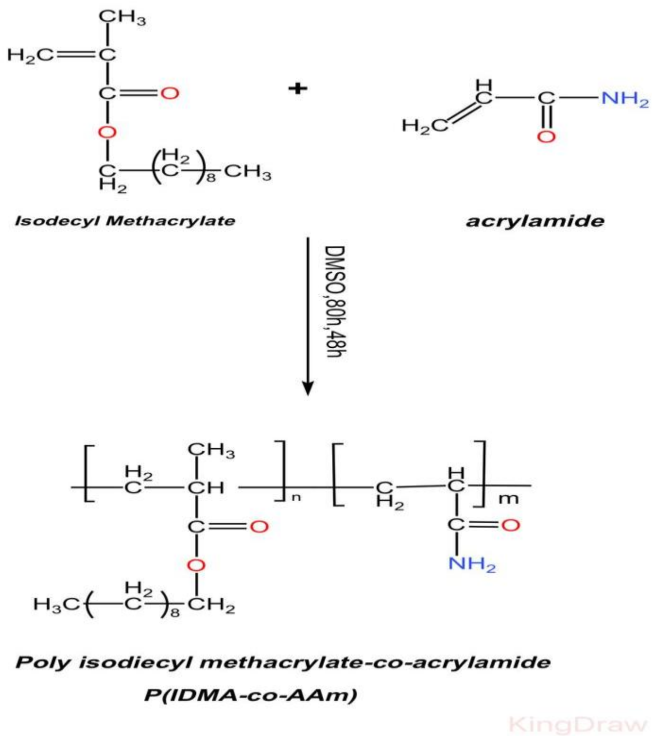


**Fig. 2** *Synthesis of IDMA/AAM copolymer*

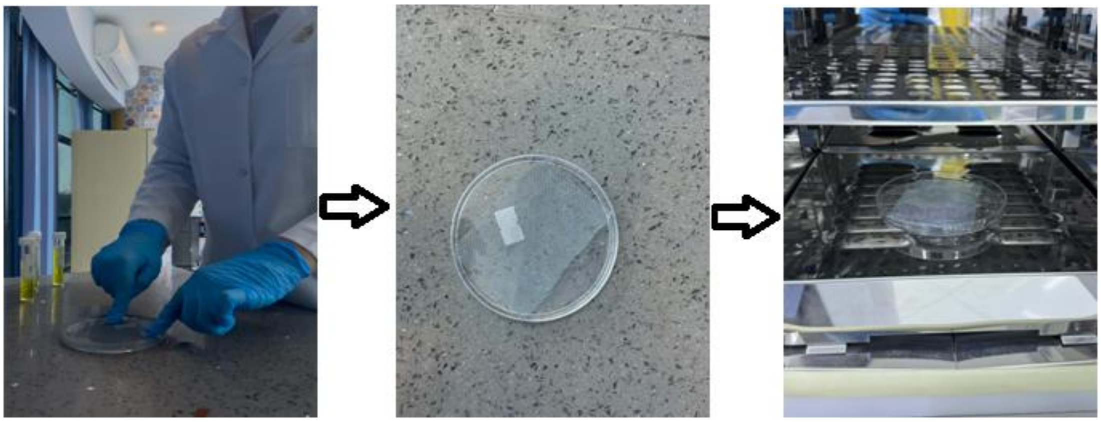

**Fig. 3** *Preparation of polymer-mesh composite*

**Separation oil from water using polymer/mesh composite**

The prepared composite was tested for oil/water separation by sandwiched them between two open stainless steel pieces. Oil/water mixture was prepared by mixing 50 ml of oil and water in graduated beaker. The oil/water mixture was gradually poured onto the top of the coated mesh. Due to the hydrophilicity of the copolymer, the water could pass through the polymer whereas oil could not pass.

Water recovery and reclaimed oil were calculated by using the following equation [16]:

$$Water\ Recovery = \frac{(Vwater)filtrate}{(Vwater)initial} \times 100 \qquad (1)$$

$$Reclaimed\ Oil = 1 - \frac{(Voil)filtrate}{(Voil)initial} \times 100 \qquad (2)$$

After separation, the amount of residual oil in the filtrated water must be determined. The filtrated water was mixed and shaken with hexane in separation funnel. The mixture was then left until forming two phases, organic and water phases. Finally, the organic phase was separated and analyzed by using UV-VIS spectrophotometer, the concentration of oil was determined by following the calibration curve.

**Results and discussion:**

Figure 4 shows the FTIR spectrum of synthesized isodecyl methacrylate IDMA. The absorption bands of IDMA are as follow: 2993 $cm^{-1}$ (alkane C-H streaching), 1720 $cm^{-1}$ (ester C=O), 1406 $cm^{-1}$ (alkane C-H bending), and 1014 $cm^{-1}$ (ester C-O). 948 $cm^{-1}$ (alkene C=C bending).

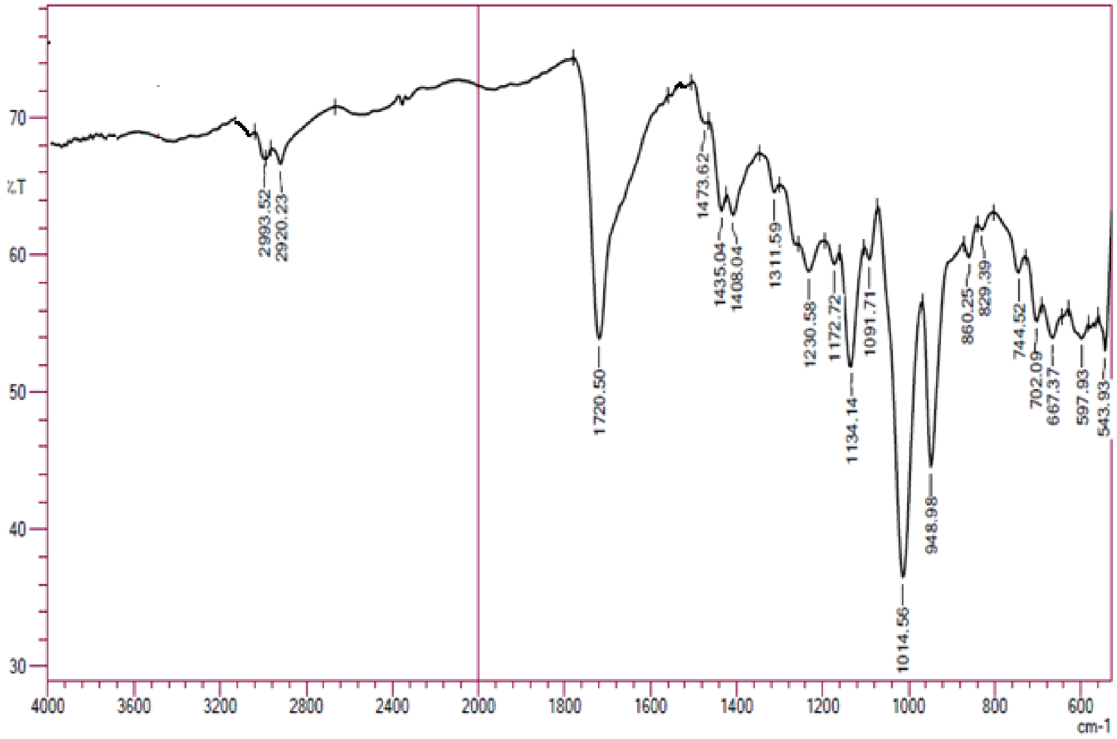


**Fig. 4** *FTIR of IDMA monomer*

$^1H$-NMR spectroscopic analysis was also employed to characterize the synthesized monomer IDMA, as shown in Figure 5. The signals at about 1.6 ppm and 4.1 ppm could be assigned to the protons of $CH_2$ in the decyl group and the protons of O-$CH_2$, respectively. The protons of the terminal –$CH_3$ group in the decyl chain group resonate at 0.79 ppm, while protons of $CH_3$ attached to the main chain backbone resonate at 1.9 ppm. The signals at 5.4 ppm and 5.9 ppm are assigned to the methylene group of the main chain backbone. The protons of $(CH_2)_9$ of the decyl groups resonate at about 1.2 ppm.

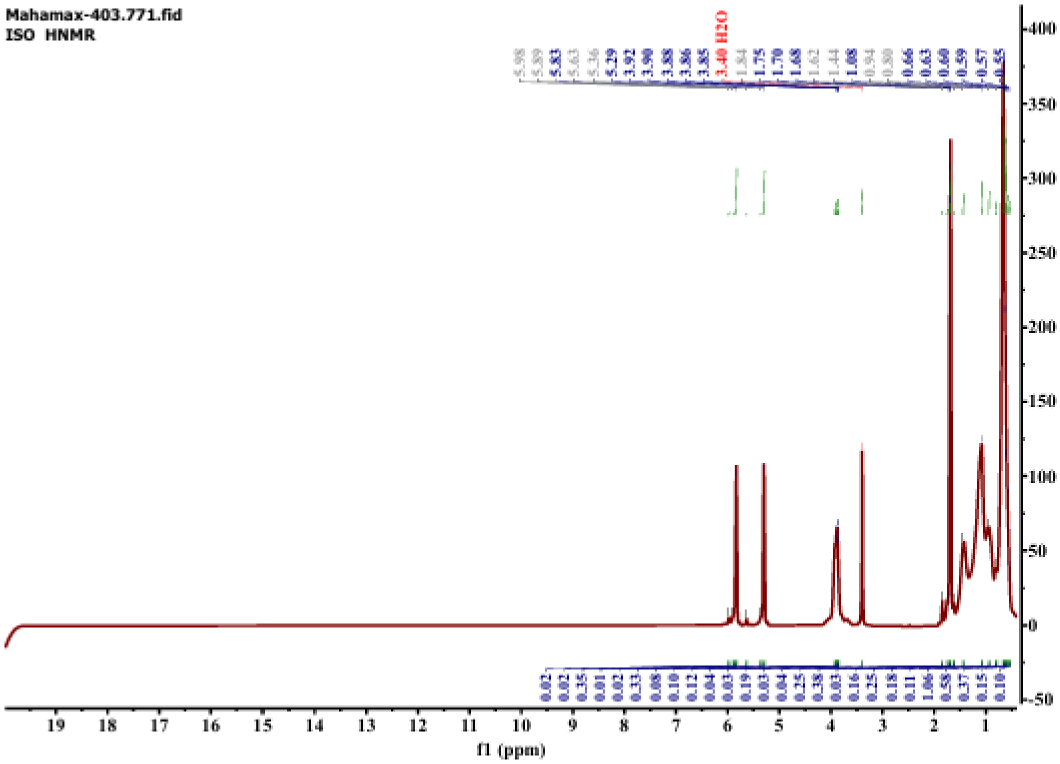


**Fig. 5** *[1]HNMR of isodecyl methacrylate*

The stretching vibration in various functional groups of the respective homopolymers is responsible for the absorption bands that show up in the FTIR spectra of the AAM/IDMA copolymers (Figure 6). The absorption bands observed are: the peak at 3340 corresponded to the N-H asymmetrical stretching vibration of the amide group in AAM, 1716 (ester C=O), 1646 (amide C=O) of AAM. The C-H bending vibration of methylene and methyl groups appears at 1495 $cm^{-1}$ and 1435 $cm^{-1}$, respectively. The C-N stretch vibration of AAM is responsible for the sharp band at 1249 $cm^{-1}$, the C-H stretching vibration is responsible for the peak at 2927 $cm^{-1}$, and the N-H stretching vibration of AAM was represented by a band at 1384 $cm^{-1}$ and C-H symmetrical stretching on $CH_2$ and $CH_3$ groups. The bands at 1014 and 1083 $cm^{-1}$ belong to C-O-C in IDMA. The disappearing of a bands at 948 $cm^{-1}$ which belong to (alkene C=C bending), and appearing of a band at 1126 which belong to (alkane C-C) supports the formation of polymers.

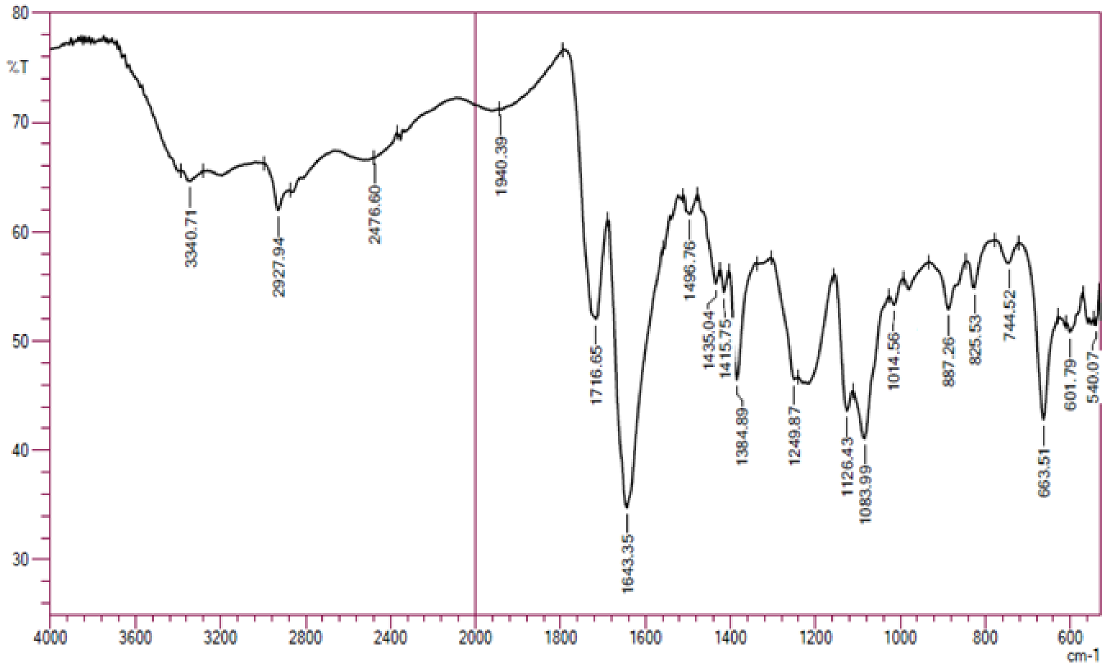


**Fig. 6** *FTIR of IDMA/AAM copolymer*

**Water Recovery**

Table 1 shows the water recovery percentage for different mesh sizes (500 and 1000 $\mu m$) based on three AAM concentrations (25, 50, and 75%) in the AAM/IDMA copolymer hydrogel-coated mesh (with and without crosslinker). It was found that when the AAM concentration in the AAM/IDMA copolymer is increased from 25% to 75%, the water recovery rises as the hydrogel thickens and the majority of the collected samples show no oil. The solutions get more viscous as the concentration of the polymer rises. In general, the two mesh sizes' water recovery ranged from 75% to 92%. Since some water is retained by the hydrogel, the water recovery did not exceed 92% due to hydrogel retention. However, the water recovery of the crosslinked polymer is lower than that of the uncrosslinked polymer. In general, a stronger gel with less water was produced by adding more EGDMA. This was anticipated since the diffusion of water molecules into the gel network was constrained by the growing crosslinker density. As a result, in the equilibrium swollen state, the hydrogel containing EGDMA absorbs less water.

**Table 1:** Water recovery of AAM/IDMA copolymer with different compositions

| Compound | EGDMA % | Water recovery of Mesh 1000 micron | Water recovery of Mesh 500 micron |
|---|---|---|---|
| 25AAM/75IDMA | 0% | 81% | 75% |
| 50AAM/50IDMA | 0% | 86% | 81% |
| 75AAM/25IDMA | 0% | 92% | 87% |
| 75AAM/25IDMA | 1% | 88% | 84% |
| 75AAM/25IDMA | 3% | 87% | 83% |

**Residual Oil in Water**

To verify the exceptional separation efficiency, UV-visible spectroscopy was used to test for the likely presence of residual oil in the filtrated water. Table 2 shows the separation efficiency percentage for different mesh sizes (500 and 1000 $\mu m$) based on three IDMA concentrations (25, 50, and 75%) in the AAM/IDMA copolymer hydrogel-coated mesh (with and without crosslinker). As the concentration of oil in the water increased, the separation efficiency decreased. This is a rise in oil pressure that allows a small amount of oil to pass through the pores. Hydrogels showed higher efficiency at lower oil concentrations. After extraction, the oil percentage in the feed and the matching filtrate (as measured with a UV-Vis spectrophotometer) were calculated. The highest separation efficiency of copolymer was for 25 % IDMA based on 1000 micron mesh which was calculated as 99% (Figure 7). Moreover, the separation efficiency has not been changed when the crosslinker was added.


**Fig. 7** *Microscopic and photographic images of oil/water mixture before A and after B separation*

**Table 2:** Separation efficiency of AAM/IDMA copolymer with different compositions

| Compound | EGDMA % | Separation efficiency of Mesh 1000 micron | Separation efficiency of Mesh 500 micron |
|---|---|---|---|
| 25AAM/75IDMA | 0% | 96% | 94% |
| 50AAM/50IDMA | 0% | 98% | 95% |
| 75AAM/25IDMA | 0% | 99% | 97% |
| 75AAM/25IDMA | 1% | 98% | 96% |
| 75AAM/25IDMA | 3% | 97% | 95% |

**Separation Duration**

In this section, the impact of mesh pore size on separation time is discussed. The separation time for a hydrogel coated mesh was shown to decrease as the mesh pores were increased for 500 and 1000 micron diameters (Table 3). 75AAM/25IDMA copolymer separated oil from water faster than 25AAM/75IDMA copolymer. In addition separation time by using AAM/IDMA hydrogel without crosslinker is less than time by using AAM/IDMA hydrogel with crosslinker; this is because the hydrogel coating on the mesh is thinner with lower concentration of monomers, leading to a quicker separation. Nevertheless, as the hydrogel thickened and its monomer concentration rose, the separation time also rose.

**Table 3:** Separation time of AAM/IDMA copolymer with different compositions

| Compound | EGDMA % | Separation time of Mesh 1000 micron | Separation time of Mesh 500 micron |
|---|---|---|---|
| 25AAM/75IDMA | 0% | 950 min | 910% |
| 50AAM/50IDMA | 0% | 789 min | 720% |
| 75AAM/25IDMA | 0% | 610 min | 593% |
| 75AAM/25IDMA | 1% | 660 min | 615% |
| 75AAM/25IDMA | 3% | 600 min | 555% |

**Mechanical properties**

Mechanical properties of prepared composites includes metallic meshes coated with polymers, with and without crosslinker, were also studied to examine the strength of coated meshes during using as membrane for oil water separation. Since 75AAM/25IDMA copolymer showed the best performance in oil/water separation, it was chosen to test its mechanical properties. Figures 8, 9, and 10 show tensile test of 75AAM/25IDMA/0EGDMA copolymer, 75AAM/25IDMA/1 EGDMA copolymer, and 75AAM/25IDMA/3 EGDMA copolymer, respectively. As it is expected, the composites including crosslinker are stronger than composites without crosslinker. The results are quite common and could be explain in term of crosslinker restricts the mobility of polymer chains, thus, the polymers becomes more strong.

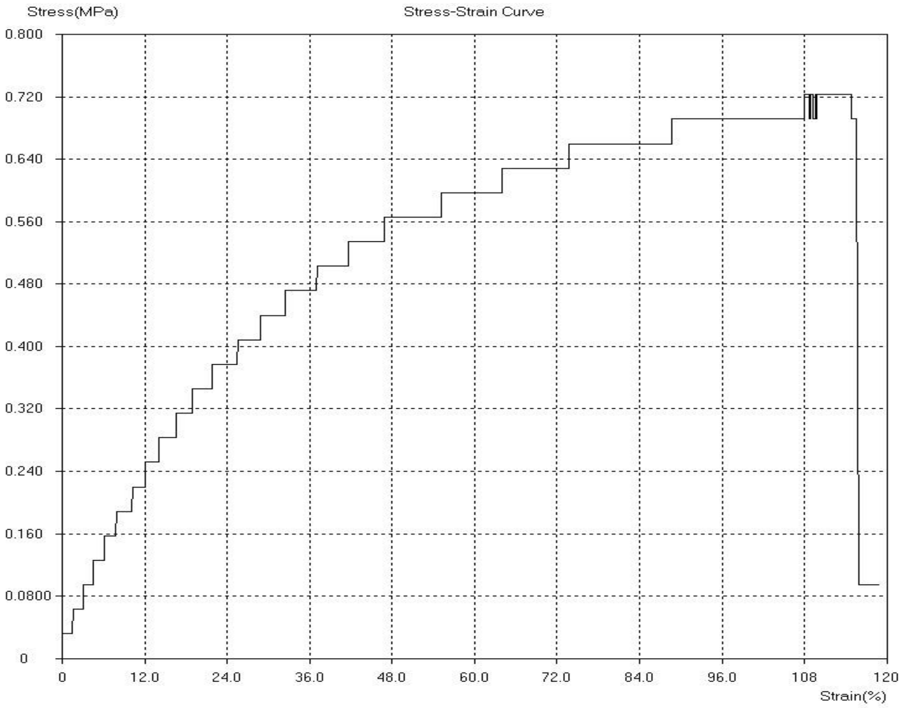


**Fig. 8** *Mechanical test of composite composed of 75AAM/25IDMA/0MBA copolymer and 1000 micron mesh*

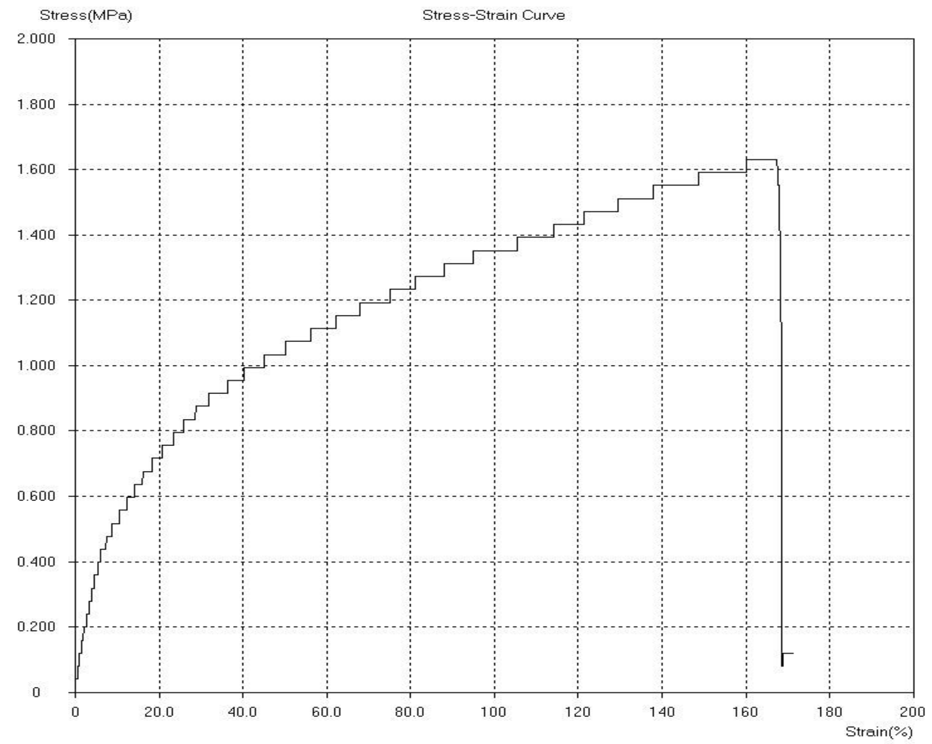


**Fig. 9** *Mechanical test of composite composed of 75AAM/25IDMA/1EGDMA copolymer and 1000 micron mesh*

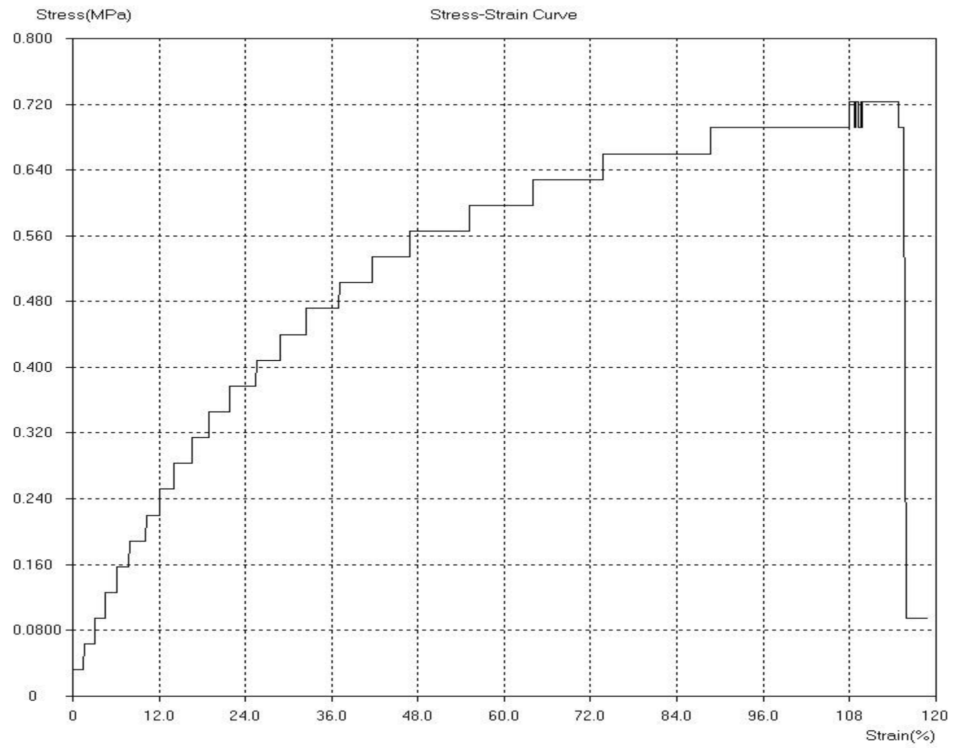


**Fig. 10** *Mechanical test of composite composed of 75AAM/25IDMA/3EGDMA copolymer and 1000 micron mesh*

## Conclusion:

After synthesis of isodecyl methacrylate monomer, its copolymer with acrylamide has been successfully achieved. The FTIR and $^1$HNMR proved the formation of monomer and copolymer. The prepared copolymers, with and without crosslinker, has been fabricated with metallic meshes, having different pores sizes, to examine their performance for separation oil from water. All Prepared composites showed good performance in oil/water separation especially when 75IDMA/25AAM copolymer was used which showed 99 % separation efficiency and 92 % water recovery. Generally, this type of copolymers can be successfully used to reduce pollution caused by different manufactories.

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# تحضير وتشخيص مونومر ايزوديسل ميثاكريلات والبوليمر المشترك له مع الاكريلامايد لفصل الزيت عن الماء

**فاطمة حكمت عسل ، امين هادي محمد**
قسم الكيمياء، كلية العلوم للبنات، جامعة بغداد
البحث مستل من رسالة ماجستير الباحث الاول

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**الكلمات المفتاحية:**
إيزوديسيل ميثاكريلات ، *البوليمر المشترك، المشبك المعدني المغطى بالهلام، فصل الزيت عن الماء، الخصائص الميكانيكية*

**معلومات المؤلف**

الايميل:
الموبايل:

**الخلاصة:**

في هذا البحث، تم تحضير مونومر إيزوديسيل ميثاكريلات IDMA عن طريق استرة حامض الميثاكريليك مع إيزوديسيل الكحول. ثم تم بلمرة مونومر IDMA مع مونومر الأكريلاميدAAM ، في تراكيب تغذية مختلفة، باستخدام تقنية بلمرة الجذور الحرة. تم تشخيص IDMA والبوليمر المشترك IDMA/AAMالمُحضر باستخدام تقنيات الرنين النووي المغناطيسي للبروتون ومطيافية الأشعة تحت الحمراء. تم تحضير مركبات من بوليمرات مُحضرة بشبكة معدنية، ذات أحجام مسام مختلفة، لاختبار أدائها في فصل الزيت عن الماء. تأثر وقت الفصل، وكمية الزيت المفصول وكمية الماء المصفى بشكل مباشر بتركيب الكوبوليمر وحجم مسام الشبكة. سُجِّلت أعلى كفاءة فصل للبوليمر المشترك بنسبة 25% من IDMA، باستخدام شبكة 1000 ميكرون، وكانت النسبة 99%. تراوحت نسبة استرداد الماء بين 75% و92%. البوليمر المشترك AAM/25IDMA75 وبدون مادة رابطة متشابكة فصل الزيت عن الماء أسرع من التركيباتالبوليمرية الأخرى. أظهرت الصور المجهرية والفوتوغرافية للماء، قبل وبعد الفصل بالبوليمرات، أن الزيت قد أُزيل تمامًا من الماء.