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Engineering and technology

Synthesis and application of nonionic graft copolymers P(mPEG-g-MMA)

^{1,2*}Myrzakhanov M.M., ^{2,3}El-Sayed Negim, ⁴Mohammad Nasir, ⁵Khaldun M. Al Azzam

¹ Satbayev University, Geology and Oil-gas Business Institute named after K. Turyssov, Department of Chemical and Biochemical Engineering, Almaty, Kazakhstan

² JSC "Kazakh-British Technical University", Almaty, Kazakhstan

³ Satbayev University, Geology and Oil-gas Business Institute named after K. Turyssov, Department of Petroleum Engineering, Almaty, Kazakhstan

⁴ University Sains Malaysia, School of Chemical Sciences, Penang, Malaysia

⁵ Pharmacological and Diagnostic Research Center (PDR), Department of Pharmaceutical Sciences, Faculty of Pharmacy, Al-Ahliyya Amman University, Amman 19328, Jordan

* Corresponding author email: m.myrzakhanov@yahoo.com

ABSTRACT

The goal of this research is to prepare novel amphiphilic graft copolymers (nonionic) based on hydrophilic poly (ethylene glycol) methyl ether (mPEG) and hydrophobic methyl methacrylate (MMA) at room temperature and pressure. To generate P(mPEG-g-MMA) grafts, poly (ethylene glycol) methyl ether (mPEG) was synthesized and grafted with varied ratios of methyl methacrylate (MMA) in the existence of benzoyl peroxide as an initiator utilizing a macro-free radical initiator procedure under the effect of heating in toluene. The research discussed in this paper looked at the possibility of using the synthesized graft copolymer P(mPEG-g-MMA) as a nonionic demulsifier in Petroleum Crude Oil Emulsions. The produced nonionic surfactants were assessed as water demulsifiers in oil emulsions that were noticeable at varying oil: water ratios at 60°C. According to the demulsifier chemical compositions as well as concentrations, the testing findings revealed that the dehydration rate of the prepared demulsifiers reached 100%. The optimal demulsifier dose was 300 ppm.

Keywords: Methyl methacrylate, Polyethylene glycol methyl ether, free radical polymerization, demulsifier, catalyst.

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Information about authors:

Myrzakhanov Maxat Makhmudovich

Ph.D. student at Satbayev University, K. Turyssov Geology and Oil-gas Business Institute, Department of Chemical and Biochemical Engineering, Almaty, Kazakhstan. Researcher at Kazakh-British Technical University, Almaty, Tole bi street, 59, Kazakhstan. Email: m.myrzakhanov@yahoo.com; ORCID ID: <https://orcid.org/0000-0001-7769-6251>

El-Sayed Negim Moussa

Ph.D., Professor of the CEC Chemical Engineering, Kazakh-British Technical University, Almaty, Tole bi street, 59, Kazakhstan. E-mail: elashmawi5@yahoo.com; ORCID ID: <https://orcid.org/0000-0002-4370-8995>

Mohamad Nasir Bin Mohamad

Ph.D., Associate professor at School of Chemical Sciences, Universiti of Sains Malaysia, 11800 USM Penang, Malaysia. Email: mnm@usm.my; ORCID ID: <https://orcid.org/0000-0002-6784-5775>

Khaldun M. Al Azzam

Ph.D., Assistant professor at Department of Pharmaceutical Sciences, Pharmacological and Diagnostic Research Center (PDR), Faculty of Pharmacy, Al-Ahliyya Amman University, Amman 19328, Jordan. Email: azzamkha@yahoo.com; ORCID ID: <https://orcid.org/0000-0003-4097-6991>

Introduction

The existence of higher than 80% water throughout the production process on gas and oil platforms is a major topic of concern for the industry. This water originates from two natural sources such as the injection and reservoir during the extraction of the hydrocarbon [1].

Emulsified water mineralization and pH. The mineralization and salt content of the aqueous phase surely have an impact on the stability and kind of oil emulsions. However, two characteristics should be highlighted: This impact is indirect, because of the production of chemical compounds

with polar oil components. Oil naphthenic acids, for example, have strong surface-active characteristics and can interact with ions such as Na⁺, K⁺, Mg²⁺, Fe³⁺, and Al³⁺ that are present in formation water. At the same time, because naphthenates K and Na are readily soluble in water, they contribute to the production of oil/water (O/W) emulsions and impair the stability of water/oil emulsions stabilized by Resinous Asphaltene substances.

Unlike Na and K salts, Mg-, Fe-, and Al- salts of naphthenic acids are more soluble in oil and can stabilize the reverse kind of Water/Oil emulsion. However, when combined with a resinous-asphaltene natural emulsifier, its impact is

weakened, most likely due to a reduction in the forces of intermolecular contact between resinous-asphaltene molecules.

As a result, all these compounds lead to considerable problems in petroleum refineries, such as corrosion in equipment and pipelines, fouling, and catalyst toxicity in the upstream facility [[2], [3]]. Water-in-oil crude oil emulsions can be found at any level of the petroleum production and processing business. They are frequently undesirable in the presence of water because they might result in high pumping costs, pipeline damage, and higher transportation costs [4].

As a result, emulsions should be separated into two phases such as water and oil [5]. Crude oil comprises aromatic molecules, alkenes, carboxylic acids, phenols, naphthenes, and other hydrocarbon chemicals. Asphaltenes are the mixture's heaviest ingredients. Asphaltenes provide a protective layer on the interfacial phase between the water and oil phases, improving the stability of the interfacial film [6].

Chemical flooding has been demonstrated to improve oil recovery, and the injection of chemicals such as surfactants helps with emulsion production [[7], [8], [9]].

As a result, emulsion formation is a difficult task for the petroleum industry. For O/W separation, many demulsifiers, such as polymeric surfactants, ionic liquids, and nanoparticles, have been produced [10].

It is well understood that oil type and salinity have a significant impact on the effectiveness of surfactants used in the industry. The authors of publications [[11], [12]] studied the influence of oil type on the performance of mixed surfactants. The synergism of combinations of anionic, nonionic, and cationic surfactants in various types of oil was examined.

Natural surfactants (asphaltenes & resins) present in the oil contribute to the stability of water-in-crude oil emulsions. These components are recognized as natural stabilizers of crude oil emulsions because they adsorb spontaneously at the water-oil interface and form an adsorption layer. Because of their interfacial activity, asphaltenes and resins are high molecular oil components that provide a large structural-mechanical barrier at the oil-water interface [[13], [14]].

Emulsion stability is the uniformity of emulsion qualities throughout time. Simultaneously, the emulsion is considered an unstable system from a thermodynamic, and emulsion features fluctuate slowly, as do several processes that occur during

emulsion property change, such as creaming, flocculation, and Ostwald ripening, coalescence, and so on. These instances, however, can occur in groups or separately [15].

To create effective and relevant techniques for oil separation, the stability of O/W emulsions has been explored in several petroleum oil research studies [16].

Already in our earlier work [17], the grafted copolymer P(mPEG-g-MMA) so produced was studied by ^1H NMR, ^{13}C NMR, FT-IR, DSC, TGA, and SEM methods.

Experimental technique

Research materials. Toluene (ACS reagent, reagent ISO, >99.7%) and hexane (HPLC grade) were purchased from Sigma Aldrich. Methyl methacrylate (MMA) (which contains 30 ppm of hydroquinone monomethyl ether (MEHQ) as an inhibitor (99%). Alfa Aesar supplied 97% (dry weight) dibenzoyl peroxide, wet with 25% water. In the experimental part, chemicals from table 1 were employed without purification.

Table 1 – Chemicals used in the synthesis

Name	Structure	Source
Polyethylene glycol methyl ether		Sigma Aldrich
Methyl methacrylate		Sigma Aldrich
Benzoyl peroxide		Alfa Aesar
Toluene		Sigma Aldrich
Hexane		Sigma Aldrich

The preparation of grafted copolymer P(mPEG-g-MMA). The synthesis of the grafted copolymer was carried out in accordance with the mechanism reported in the reference [18].

The grafted copolymers in various ratios were synthesized in a 500 ml Syrris Globe reactor with a condenser and constant stirring.



Figure 1 – Syrris Globe reactor used for synthesis with doser

The first step was to dissolve mPEG in toluene, followed by the addition of a catalyst (0.1% weight of monomer). In free radical polymerization, benzoyl peroxide was utilized as a catalyst. Benzoyl peroxide was recrystallized, then dried in an oven at temperature of less than 400°C and later in a desiccator over silica gel to get rid of the water content. The monomer (MMA) was injected into the solution drop by drop in the second stage, and the temperature was increased to 700°C for one hour. Polymerization of mPEG with methyl methacrylate in various ratios, such as 10:90, 20:80, and 30:70, was carried out. A thermometer was used to record the temperature, and the stirring speed was set at around 100 rpm. For the last stage, the temperature was raised to 850°C for 1 hour. The reaction product was then precipitated out using hexane. The precipitated copolymer was filtered and then dried overnight at room temperature. The final dry products were powders.

Table 2 – Composition of grafted copolymers

Name	Sample 1, g	Sample 2, g	Sample 3, g
PGME	5	10	15
MMA	45	40	35
Catalyst	0.05	0.1	0.15
Toluene	100	100	100
Hexane	100	100	100



Figure 2 – Synthesized P(mPEG-g-MMA) copolymers

Preparation of Water/Oil Emulsions. All the emulsions were made in a total volume of 100 ml. The crude oil-to-aqueous phase (sea water) ratio was raised from 10-50%. (Vol.%). The emulsions were made by combining them with an IKA T25 digital ULTRA-TURRAX homogenizer. The speed was set to 1500 rpm for one hour.

The prepared emulsion was put into graduated 100 ml beakers. Starting with the second test flask, the determined amount of demulsifier was added and the emulsion was agitated for 1 minute. The emulsion was then allowed to stand for 30 minutes at the demulsifier's operating temperatures. The water, in the first test flask, was allowed to stand without any demulsifier. After that, the amount of water was measured with an accuracy of 0.1 ml. The demulsifier's effectiveness was determined by dividing the volume of water released by the total amount of water in the emulsion.

In this regard, the crude oil was stirred in a 100 mL beaker at 350°C (1500 rpm) while water was slowly combined with the oil until the two phases were completely homogeneous. At various oil :water ratios, the emulsions were noticeable (90 :10, 80 :20, 70 :30, and 50 :50).



Figure 3 – Emulsion prepared in different ratios of oil and water

Methods and techniques for characterization of emulsifier efficiency

The international standard "Bottle-Test" was used to assess the efficiency of new grafted copolymer reagent-demulsifiers. The bottle test technique is utilized in the production site to identify demulsifiers by calculating water separation performance on crude oil emulsions [19].

The tests involve determining the dynamics of oil-water emulsion stratification in time as a result of reagent-demulsifier and temperature action, followed by determining the residual water and chloride salts content.

At the dehydration step, we placed 70ml of oil in a 100ml graduated test tube, carefully capped with a cork, and placed in the thermostat for 5-7 minutes to heat up to 60°C.

Adding reagent-demulsifier in the required dosage (ppm), shaking vigorously for 1.5 minutes, and placing in thermostat to determine the dynamics of oil-water emulsion stratification in time -0.5, 1, and 2 hours at 60°C: the volume of the stratified water phase was measured in time.

After 60 minutes, the bottle test produced approximately 50% water separation at a demulsifier dosage of 300 ppm.

The method [20] was used to determine the residual water content of an oil phase sample.

The full volume of the aqueous phase was separated and discarded for the next step of testing.

Research Characterization

Differential scanning calorimetry (DSC), thermal gravimetric analyses (TGA), scanning electron microscopy (SEM), nuclear magnetic resonance (^1H NMR & ^{13}C NMR), and Fourier transform infrared FT-IR spectroscopies are used to evaluate the graft copolymers P(mPEG-g-MMA).

Research Results and Discussion

The grafted copolymer P(mPEG-g-MMA) was effectively made by free radical polymerization utilizing benzoyl peroxide as an initiator. SEM, FT-IR, ^1H NMR, ^{13}C NMR, TGA, and DSC were all used to characterize the samples. NMR analysis revealed the presence of all groups in the copolymers. According to the thermal stability investigations, the grafted copolymer has outstanding thermal stability and may be employed in a variety of applications. Furthermore, grafting and polymerization of the grafted samples greatly enhance the rate of breakdown. As a result, this copolymer has a wide range of uses, including surfactants in chemical technology.

Table 3 – Effectiveness of demulsifiers on needed time to separate oil from water at room temperature

Name	Amount, ppm	Time, min
P(MPEG-g-MMA) 10:90	100	18
P(MPEG-g-MMA) 10:90	200	14
P(MPEG-g-MMA) 10:90	300	9
P(MPEG-g-MMA) 10:90	400	8
P(MPEG-g-MMA) 10:90	500	5



Figure 4 – End of oil-water emulsion demulsification process by grafted copolymer

Three different demulsifier ratios were examined for the performance of water separation at 30°C using the bottle-test experiment methods outlined in the preceding section. The oil :water ratio was chosen such as the emulsion sample, 70 :30, and P(MPEG-g-MMA) was chosen as the reagent demulsifier, 10 :90. The results were collected for a 100 mL emulsion at 250°C and atmospheric pressure. The maximum demulsifier dose is established by the tests with the purpose of the optimizing demulsifier dosage to optimize water separation. As indicated in table 3, the reagent-demulsifier with a concentration of 300 ppm produced the best results compared to the others. All three demulsifiers produced excellent water, with no oil droplets on the bottle/tube wall and clear, colorless water.

Conclusions

The grafted copolymer P(mPEG-g-MMA) was effectively produced by free radical polymerization utilizing benzoyl peroxide as an initiator. SEM, FT-IR,

nuclear magnetic resonance (¹³C NMR & ¹H NMR), TGA, and DSC were all used to characterize the samples. NMR analysis revealed the presence of all groups in the copolymers. Based on the thermal stability outcomes, the grafted copolymer has good thermal stability and thus might be used as a surfactant-demulsifier in the Crude Oil Petroleum business. It has been established that the proper dose will have an economic effect.

Conflict of interest. All authors declare that there is no conflict of interest.

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P(mPEG-g-MMA) иондық емес егілген сополимерлердің синтезі және қолданылуы

^{1,2} Мырзаханов М.М., ^{2,3} Эль Сайд Негим, ⁴ Мохаммад Насыр, ⁵Халдун М. Аль Аззам

¹ Сәтбаев Университеті, Қ. Тұрысов атындағы Геология және мұнай-газ ісі институты, Химиялық және биохимиялық инженерия кафедрасы, Алматы, Қазақстан Республикасы

² АҚ “Қазақстан - Британ Техникалық Университеті”, Алматы, Қазақстан Республикасы

³ Сәтбаев Университеті, Қ. Тұрысов атындағы Геология және мұнай-газ ісі институты, Мұнай инженериясы кафедрасы, Алматы, Қазақстан Республикасы

⁴ Сайнс Малайзия Университеті, Химия ғылымдары мектебі, Пенанг, Малайзия

⁵ Фармакологиялық және диагностикалық зерттеу орталығы (PDRC), Фармацевтикалық ғылымдар бөлімі, Фармацевтика факультеті, Амман университеті Әл Ахлия, Амман, 19328, Иордания

ТҮЙІНДЕМЕ

Бұл жұмыстың мақсаты қоршаған орта температурасында және қалыпты атмосфералық қысымда гидрофильді поли(этиленгликоль) метил эфирі (MPEG) және гидрофобты метилметакрилат (MMA) негізіндегі жаңа иондық емес амфифильді сополимерлерін алу болып табылады. Поли(этиленгликоль) метил эфирі (mPEG) әртүрлі қатынаста 10:90, 20:80, 30:70 метилметакрилат (MMA) дайындалды және инициатор ретінде бензоил асқын тотығының қатысуымен жылу астында макробосрадикалды инициатор әдісімен егілді. P(mPEG-g-MMA) сополимерлерін алу үшін толуолда. Осы жұмыста ұсынылған зерттеулер мұнай эмульсиялары үшін иондық емес дезэмульгатор ретінде синтезделген трансплантаттық сополимер P(mPEG-g-MMA) мүмкін пайдалану мақсатында жүргізілді. Дайындалған иондық емес беттік белсенді заттар 60°C температурада әртүрлі мұнай: су

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	<p>қатынасында көрсетілген майдағы су эмульсиялары үшін деэмульгаторлар ретінде бағаланды. Тәжірибе нәтижесі дайындаған деэмульгаторлардың сусыздану жылдамдығы деэмульгаторлардың химиялық құрамы мен концентрациясына байланысты 100%-ға жететінін көрсетті. Деэмульгатордың оңтайлы дозасы 300 ppm болды.</p> <p>Түйін сөздер: полиэтиленгликоль метил эфиірі, метилметакрилат, бос радикалды полимерлеу, катализатор, деэмульгатор.</p>
Мырзаханов Максат Махмудович	<p>Авторлар туралы ақпарат: PhD докторант, Сәтбаев Университеті, Қ. Тұрысов атындағы Геология және мұнай-газ ісі институты, Химиялық және биохимиялық инженерия кафедрасы; Ғылыми қызметкер, «Қазақстан-Британ техникалық университеті» АҚ, Алматы қ., Қазақстан. Email: m.myrzakhanov@yahoo.com; ORCID ID: https://orcid.org/0000-0001-7769-6251</p>
Эль-Сайд Негим Мусса	<p>PhD докторы, «Химиялық инженерия» РЭК профессоры, «Қазақстан-Британ техникалық университеті» АҚ, Алматы қ., Қазақстан. E-mail: elashmawi5@yahoo.com; ORCID ID: https://orcid.org/0000-0002-4370-8995</p>
Мохаммад Насыр	<p>PhD, Малайзия ғылым университетінің профессоры, Пенанг, Малайзия. Email: mnm@usm.my; ORCID ID: https://orcid.org/0000-0002-6784-5775</p>
Халдун М. Аль Аззам	<p>PhD, доцент, Фармацевтикалық ғылымдар кафедрасы, Фармакологиялық және диагностикалық зерттеу орталығы (PDRC), Фармация факультеті, Амман Әл-Ахлия университеті, Амман, 19328, Иордания, Email: azzamkha@yahoo.com; ORCID ID: https://orcid.org/0000-0003-4097-6991</p>

Синтез и применение неионогенных прививочных сополимеров P(mPEG-g-MMA)

^{1,2} Мырзаханов М.М., ^{2,3} Эль Сайд Негим, ⁴ Мохаммад Насыр, ⁵Халдун М. Аль Аззам

¹ Сәтбаев Университет, Институт геологии и нефтегазового дела им. К. Турысова, Кафедра химической и биохимической инженерии, Алматы, Казахстан

² АО «Казахстанско-Британский технический университет», Алматы, Казахстан

³ Сәтбаев Университет, Институт геологии и нефтегазового дела им. К. Турысова, Кафедра нефтяной инженерии, Алматы, Казахстан

⁴ Университет Сайнс Малайзия, Школа химических наук, Пенанг, Малайзия

⁵ Центр фармакологических и диагностических исследований (PDRC), отделение фармацевтических наук, фармацевтический факультет, Амманский университет Аль-Ахлия, Амман 19328, Иордания

АННОТАЦИЯ

Цель данной работы - получение новых неионогенных амфифильных прививочных сополимеров на основе гидрофильного поли(этиленгликоль) метилового эфира (МПЭГ) и гидрофобного метилметакрилата (ММА) при температуре окружающей среды и нормальном атмосферном давлении. Поли(этиленгликоль) метиловый эфир (mPEG) был подготовлен и привит с различными соотношениями 10:90, 20:80, 30:70 метилметакрилата (ММА) в присутствии пероксида бензоила в качестве инициатора с использованием метода макросвободнорадикального инициатора под воздействием нагревания в толуоле для получения сополимеров P(mPEG-g-MMA). Исследования, представленные в данной работе, проводились с целью возможного применения синтезированного прививочного сополимера P(mPEG-g-MMA) в качестве неионогенного деэмульгатора для нефтяных эмульсий. Приготовленные неионогенные ПАВ были оценены как деэмульгаторы для эмульсий "вода в нефти", которые были выражены при различных соотношениях нефть: вода при температуре 60°C. Результаты эксперимента показали, что скорость дегидратации приготовленных деэмульгаторов достигает 100% в зависимости от химического состава и концентрации деэмульгаторов. Оптимальная дозировка деэмульгатора составила 300 ppm.

Ключевые слова: метиловый эфир полиэтиленгликоля, метилметакрилат, свободнорадикальная полимеризация, катализатор, деэмульгатор.

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Мырзаханов Максат Махмудович

Информация об авторах:

PhD студент, Сәтбаев Университет, Институт геологии и нефтегазового дела им. К. Турысова, Кафедра химической и биохимической инженерии, Алматы, Казахстан. Научный сотрудник, АО «Казахстанско-Британский Технический Университет», Алматы, Казахстан. Email: m.myrzakhanov@yahoo.com; ORCID ID: <https://orcid.org/0000-0001-7769-6251>

Эль-Сайед Негим Мусса	<i>PhD, профессор в НОЦ «Химическая инженерия», АО «Казахстанско-Британский технический университет», Алматы, Казахстан. E-mail: elashmawi5@yahoo.com; ORCID ID: https://orcid.org/0000-0002-4370-8995</i>
Мохамад Насыр	<i>PhD, ассоциированный профессор, «Университет Сайнс Малайзия», Пенанг, Малайзия. Email: mnm@usm.my; ORCID ID: https://orcid.org/0000-0002-6784-5775</i>
Халдун М. Аль Аззам	<i>Доктор философии, доцент кафедры фармацевтических наук, Центр фармакологических и диагностических исследований (PDRC), фармацевтический факультет, Амманский университет Аль-Ахлия, Амман 19328, Иордания, Email: azzamkha@yahoo.com; ORCID ID: https://orcid.org/0000-0003-4097-6991</i>

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