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SYNTHESIS OF RUBBER-BASED HYDROPHOBIC OLEOGELS AND THE STUDY OF SORPTION-REGENERATION PROCESSES IN THE TREATMENT OF OILY WASTEWATER

Xusanova Mamlakat Furqatovna,
t.f.f.d. katta ilmiy xodim
mamlakatkhusanova@gmail.com
https://orcid.org/0009-0005-3845-677X
Tashkent Scientific Research Institute of Chemical Technology,

Abstract: *Hydrophobic, mechanically durable, and reusable sorbents are required for removing oily water wastes from petrochemical plants, transport systems, mechanical shops, and industries. This study synthesizes rubber oleogels based on the ternary system of SBR-1502, SKI-3 NST, and SMR L rubbers. Oleogels were prepared in aromatic solvents (toluene - OG-T and m-xylene-OG-X) using benzoyl peroxide as the radical initiator and hexamethylenediamine as the copolymerization agent. The materials were characterized by FTIR spectrometry, scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX). FTIR data have shown the retention of the hydrocarbon chain of the polyisoprene and butadiene and participation of aromatic regions in network formation. Microstructural analysis by SEM has revealed a porous three-dimensional structure. According to EDX results, carbon dominated in the chemical composition of the samples as 82.56 wt.% C, while oxygen occupied 17.44 wt.% O, providing evidence of hydrophobic and oleophilic properties. Thermal analysis has suggested multi-stage decomposition with maximum weight loss between 390 and 400 °C.*

Keywords: *oily wastewater; technogenic wastewater; rubber-based oleogel; hydrophobic sorbent; oil-water separation; civil engineering; thermal stability; mechanical properties; adsorption kinetics; regeneration.*

KAUCHUK ASOSIDAGI GIDROFOB OLEOGELLARNING SINTEZI QILISH VA MOYLI OQOVA SUVLARNI TOZALASHDAGI SORBSION-REGENERATSION JARAYONLAR O‘ RGANISH

Annotatsiya: *Neftkimyo zavodlari, transport tizimlari, mexanik sexlar va sanoat korxonalaridan yog‘ li suv chiqindilarini olib tashlash uchun gidrofobik, mexanik jihatdan chidamli va qayta ishlatiladigan sorbentlar talab etiladi. Ushbu tadqiqotda SBR-1502, SKI-3 NST va SMR L rezinalarining uchkomponentli tizimiga asoslangan rezina oleogellari sintez qilingan. Oleogellar aromatik erituvchilarda (toluen OG-T va m-ksilen OG-X) benzoyl peroksidni radikal initsiator, geksametilendiaminni esa kopolimerlash agenti sifatida ishlatib tayyorlandi. Materiallar FTIR spektrometriyasi, energiya disperziyali rentgen spektroskopiyali skanerlangan elektron mikroskopiya (SEM/EDX), sinovlari bilan xarakterlandi. FTIR ma’lumotlari poliizoprendagi va butadiendagi uglevodorod zanjirining saqlanib qolganini hamda aromatik sohalarning tarmoq hosil bo‘ lishida ishtirok etishini ko‘ rsatdi. SEM yordamida o‘ tkazilgan mikrostrukturali tahlil porali uch o‘ lchovli tuzilmani aniqladi. EDX natijalariga ko‘ ra, namunalarning kimyoviy tarkibida uglerod 82,56 vazn foizini, kislorod esa 17,44 vazn foizini tashkil etib, bu gidrofobik va oleofilik xususiyatlarga ega ekanligini ko‘ rsatdi. Termik tahlil ko‘ p bosqichli parchalanishni ko‘ rsatdi, maksimal vazn yo‘ qotilishi 390-400 °C oralig‘ ida sodir bo‘ ladi.*

Kalit so‘ zlar: *moyli chiqindi suv; texnogen chiqindi suv; rezina asosidagi oleogel; gidrofobik sorbent; moy-suv ajratish; fuqarolik muhandisligi; issiqlik barqarorligi; mexanik xususiyatlar; adsorbsiya kinetikasi; regeneratsiya*

СИНТЕЗ ГИДРОФОБНЫХ ОЛЕОГЕЛЕЙ НА ОСНОВЕ КАУЧУКА И ИЗУЧЕНИЕ СОРБЦИОННО-РЕГЕНЕРАЦИОННЫХ ПРОЦЕССОВ ПРИ ОЧИСТКЕ НЕФТЕСОДЕРЖАЩИХ СТОЧНЫХ ВОД

Аннотация: *Для очистки сточных вод, загрязнённых нефтепродуктами, на нефтехимических заводах, в транспортных системах, механических цехах и других промышленных предприятиях требуются гидрофобные, механически прочные и многоразовые сорбенты. В данном исследовании синтезированы каучуковые олеогели на основе тройной системы каучуков SBR-1502, SKI-3 NST и SMR L. Олеогели получали в ароматических растворителях (толуол OG-T и м-ксилол OG-X) с использованием перекиси бензоила в качестве радикального инициатора и гексаметилендиамина в качестве агента сополимеризации. Характеристики материалов определяли с помощью ФУИР-спектрометрии, сканирующей электронной микроскопии с энергодисперсионной рентгеновской спектроскопией (СЭМ/ЭДРС). Данные ФУИР-спектроскопии показали сохранение углеводородной цепи полиизопрена и бутадиена, а также участие*

ароматических участков в формировании сети. Микроструктурный анализ с помощью СЭМ выявил пористую трехмерную структуру. Согласно результатам ЭДХ, в химическом составе образцов преобладал углерод (82,56 мас. % C), тогда как кислород составлял 17,44 мас. % O, что свидетельствует о гидрофобных и олеофильных свойствах. Термический анализ показал многоступенчатое разложение с максимальной потерей массы в диапазоне температур от 390 до 400 °C.

Ключевые слова: *нефтесодержащие сточные воды; техногенные сточные воды; олеогель на основе каучука; гидрофобный сорбент; разделение нефти и воды; гражданское строительство; термическая стабильность; механические свойства; кинетика адсорбции; регенерация.*

Introduction

Oily wastewater represents a challenging issue in petrochemical plants, motor vehicle filling stations, port terminals, metal processing workshops, runoff from roads, and industrial zones [1,2]. Such wastes may contain free oil, droplets of dispersed oil, emulsified oil, surfactants, suspended matter, and even heavy metals or other refractory pollutants [3]. In this regard, the multicomponent nature of oily effluents complicates their purification as a material or process capable of removing free oil might be ineffective in treating dispersed oil or emulsion stabilized with surfactants [1,4]. From a civil engineering standpoint, this issue is significant for stormwater pretreatment, drainage, wastewater polishing prior to disposal, and oil spill cleanup [5,6].

The environmental importance of oily and technogenic wastewater goes far beyond visible pollution. Oil films in water bodies hinder gas exchange in the air/water boundary layer leading to oxygen depletion and disruption of natural wastewater self-purification [8]. At the same time, dissolved and dispersed oil components, e.g., aromatic hydrocarbons, may pose toxic, mutagenic, and long-term harmful effects to aquatic microorganisms and biota [9]. Continuous or periodic effluent discharge results in biodiversity reduction, imbalance in ecosystems, and hazardous substance accumulation in bottom sediments and soils [10]. Wastewater produced by oil extraction and processing (produced water) is particularly problematic due to high volume and varying composition depending on crude oil origin, chemical additives, processing technology, and salinity of the water [11]. In addition, the content of contaminants in produced water may change significantly with time affecting the performance of separation technologies [12]. Recent reviews show that exposure to produced-water contaminants can have negative effects on benthic community, aquatic organism early stages, and overall marine ecosystem functioning [13]. This trend adds to an increased environmental risk posed by the discharge of oily wastewater, driven by the implementation of strict effluent quality requirements and recycling of industrial wastewater [14]. Thus, treatment of oily wastewater is no longer regarded as an end-of-pipe operation but becomes an integral element of water and environmental engineering [15].

Many different technologies have been proposed for wastewater oil removal, including gravitational separation, flotation, coagulation/flocculation, membrane filtration, adsorption, electrocoagulation, advanced oxidation processes, and hybrid processes [16,17]. These methods are characterized by a different separation principle, efficiency, costs, and applicability to the oil stabilization degree, presence of dissolved organics, salt concentration, and size of colloidal particles [18]. Conventional physical methods, e.g., gravitational sedimentation, have the advantage of simplicity and low energy demand but are primarily effective for free oil removal [19]. Coagulation and flocculation are commonly applied as pretreatment technologies since they destabilize oil emulsions and enhance separation [20]. Membrane processes including ultrafiltration, reverse osmosis, and membrane distillation have proven highly efficient in wastewater polishing but suffer from issues with fouling, wetting, and cleaning [21]. Electrocoagulation combines coagulation, flotation, and electrochemical processes and received growing interest despite challenges related to passivation of electrodes and energy costs [22]. Advanced oxidation processes stand out in wastewater treatment as efficient in mineralization of refractory dissolved organics, although large-scale plants still face problems with oxidant consumption and cost-effectiveness [23]. Adsorption is considered one of the most flexible separation technologies due to relative simplicity and high affinity to hydrophobic pollutants [24]. However, traditional adsorbents can show poor efficiency in multicomponent systems and limited regenerability [25].

Materials and Methods

Materials

The rubber phase consisted of a ternary blend of SBR-1502 styrene-butadiene rubber, SKI-3 NST isoprene rubber, and SMR L natural rubber. The SBR-1502 was the commercially available form of butadiene-styrene rubber; the SKI-3 NST was the form of the isoprene rubber; and the SMR L was the natural rubber component. Benzoyl Peroxide (BPO; CAS 94-36-0; Purity 97%) was used as the radical initiator. Hexamethylenediamine (HMDA; CAS 124-09-4) was used as the amine crosslinking/co-curative. Toluene (CAS 108-88-3) and m-Xylene (CAS 108-38-3; Purity 99%) were used as the aromatic

solvent. All chemicals were used as received without further purification. The synthesis procedure took place in a two-neck round-bottom flask fitted with a mechanical stirrer and a temperature controller.

Table 1.

Chemicals Used in Synthesis of Oleogel Based on Rubber

Component	Specification	Function in oleogel synthesis
Butadiene-styrene rubber	SBR-1502	Hydrophobic elastic rubber component, which adds styrenic aromatic groups and butadiene unsaturated components to the formulation.
Isoprene rubber	SKI-3 NST	Hydrocarbon elastic rubber compound that offers polyisoprene groups.
Natural rubber	SMR L	Elastic rubber compound enriched with polyisoprene.
Benzoyl peroxide	0.20 g per batch; CAS 94-36-0; 97% purity	Initiator for thermal curing.
Hexamethylenediamine	2.00 g per batch; CAS 124-09-4	Crosslinking compound/curing agent.
Toluene	90.0 g per OG-T batch; CAS 108-88-3	Aromatic solvent and network forming medium.
m-Xylene	90.0 g per OG-X batch; CAS 108-38-3; 99% purity	Aromatic solvent and network-forming medium

Formulations of oleogels

The two oleogel samples made included OG-T, the toluene oleogel, and OG-X, the m-xylene oleogel. In each oleogel formulation, the total amount of rubber added was 10.0 g and the solvent weight was 90.0 g, making up a 10 wt% rubber solution before crosslinking. The rubber mix consisted roughly of 1/3 of each type of rubber component; the exact masses were as follows: 3.33 g of SBR-1502, 3.33 g of SKI-3 NST, and 3.34 g of SMR L.

Table 2.

Composition of rubber oleogels by batch.

Sample code	SBR-1502 (g)	SKI-3 NST (g)	SMR L (g)	Total rubber (g)	Solvent and mass	BPO (g)	HMDA (g)
OG-T	3.33	3.33	3.34	10.00	Toluene, 90.0 g	0.20	2.00
OG-X	3.33	3.33	3.34	10.00	m-Xylene, 90.0 g	0.20	2.00

Preparation of rubber-based oleogels

Oleogels were prepared using a two-neck round bottom flask equipped with a mechanical stirrer and a thermometer. In a first step, the rubber compound was dissolved in the selected solvent (toluene in the case of OG-T, and m-xylene in the case of OG-X), producing a 10 wt% rubber solution. The mixture was heated to 60 °C and stirred for 30 min. The temperature was increased to 80 °C, and 0.20 g benzoyl peroxide was added. After dispersing the initiator in the reaction media for 60 s, 2.00 g hexamethylenediamine was added. The temperature was kept at 85 °C during 2 h of reaction time with a stirring rate of 100 rpm. Once a dark orange color appeared, suggesting the creation of the network, the temperature was lowered to 70 °C, and kept constant for 4 more hours to ensure the maturing of the network. To remove volatile residues from the product, the oleogel was dried in a muffle furnace at 40 °C during 24 h. The dry masses obtained for both OG-T and OG-X and their yields were obtained from the lab notebook and are reported in the following section (OG-T, 8.45 g, 84.5% yield; OG-X, 8.12 g, 81.2% yield).

Characterization methods

The FTIR spectroscopic analyses were performed by using an IRTracer-100 spectrophotometer (Shimadzu Corporation, Japan, 2017). The spectra were obtained at a wavelength range of 4000-450 cm^{-1} at a resolution of 4 cm^{-1}. The FTIR instrument provides good signal-to-noise ratio and hence can be used for detection of weak peaks in polymer

materials. FTIR was applied to analyze aliphatic C-H stretching, C=C vibrations, aromatic peaks, and possible surface hydroxyls contribution. Spectra were baseline corrected and normalized for comparative study purposes.

The SEM measurements were carried out using a JEOL JSM-IT210 instrument. Micrographs were obtained by an accelerating voltage of 3.0 kV, which is a suitable operating voltage for analyzing fragile polymeric material without causing any surface damage. SEM images were obtained at 180x, 300x, and 500x magnifications for evaluating the surface roughness, pore structures, networking, and heterogeneity of the sample. EDX analysis (in combination with SEM) was used to obtain the elemental composition at an accelerating voltage of 15.0 kV and a working distance of 10 mm. The quantification of EDX spectra was made using ZAF correction method.

For determining mechanical tensile properties, samples in the form of dumbbells (Type IV, ASTM D638) were tested by using a universal tester with crosshead speed of 50 mm min^{-1}. Tension data were used for comparing the flexibility and stiffness of OG-T and OG-X. The major mechanical properties of interest were elongation at break (εb), tensile strength at break (σb), and modulus of elasticity (E). At least three replicates for each material type were tested. Results are shown as mean ± standard deviation.

Sorption experiments

Systematic batch sorption experiments were carried out to estimate sorption capacity, efficacy of oil removal and reuse capability of the produced oleogels under controlled laboratory conditions. All sorption measurements were performed in triplicate, and the results are presented as mean ± SD.

Preparation of model oily wastewater

Oil-in-water emulsions were prepared using a known quantity of oil product (pump oil or diesel fuel) mixed with distilled water in glass flasks with magnetic stirring (300 rpm) during 30-60 min. Wastewater samples collected from the site were either used in their original form or previously filtrated from coarse suspensions. Initially, the concentration of the oil product in the wastewater was measured by gravimetry or spectrophotometry prior to any sorption tests.

Experimental batch sorption procedure

Batch sorption experiments were conducted in 250 mL glass flasks with a constant amount (V) of polluted water (50-100 mL) and the known mass (W) of dried oleogel sample. The flask was tightly closed and put on the laboratory shaker rotating at 150 rpm. All tests were carried out at 25 ± 2 °C, except stated otherwise. First of all, the sample weight (W_0) was measured to calculate its initial mass. Afterward, the sample was immersed in the test medium and held in contact for a certain time period required to achieve sorption equilibrium. Then, the sample was taken out of the solution, and excess surface liquid was drained during 30 s. Finally, the weight of the sample (W_1) was registered again to estimate the final weight.

Influence of operational parameters

The influence of contact time was estimated by changing the time of sorbent and water contact from 5 min up to 180 min with step equal to 10 min. The effect of sorbent dosage was investigated using different masses of oleogel (0.05-0.50 g) at constant volume (100 mL) and pollution level of wastewater (initial oil concentration). Impact of the initial pollutant concentration was estimated using wastewater solutions with concentration varying within 100-1000 mg/L. The pH value of the wastewater was modified in the range of 2.0-10.0 by adding small quantities of dilute hydrochloric (0.1M) or sodium hydroxide (0.1M) solutions to the solution. Thermosensitivity of the sorption process was studied in terms of wastewater temperature (20, 30, and 40°C).

Tests for sorbents regeneration and reusability

To investigate oleogel reusability potential, regeneration tests were performed by compression of the saturated oleogel to remove absorbed oil, and subsequent washing with n-hexane. After washing, regenerated oleogel was dried at 40 °C during 24 h and subjected to new sorption test.

Results and Discussion

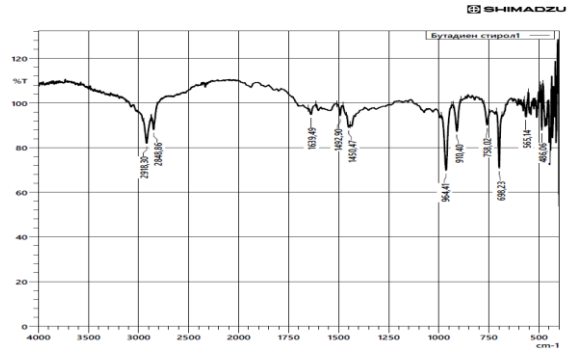
FTIR-based structural interpretation

The FTIR spectra of the rubbers used as precursors and those of the synthesized oleogels (Figure 1) show that the hydrocarbon backbone remains intact even after gel formation. In the case of the butadiene-styrene rubber precursor, aliphatic C-H stretching vibration is observed at 2913-2843 cm^{-1}, aromatic C=C vibration at 1639 and 1492 cm^{-1}, and vibrations of trans-1,4-butadiene and vinyl at 966 and 910 cm^{-1}. This confirms that the material possesses styrenic and butadienic functionalities useful for forming polymer networks via radical coupling.

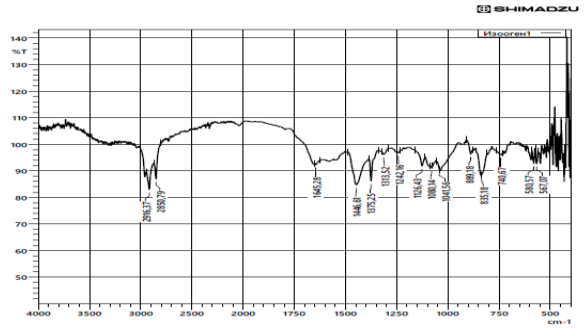
In the cases of isoprene and natural rubbers, C-H stretching is found between 2960 and 2850 cm^{-1}, and C=C stretching at 1645 cm^{-1}, which are characteristics of polyisoprene-based materials. Vibrations in the range of 1446-1375 cm^{-1} further validate the existence of methylene and methyl moieties in the rubber molecules. Thus, these vibrations confirm that the gelation process has not destroyed the backbone of the rubber molecules.

Similar to the spectra of pure polyisoprenes, the OG-X spectrum includes the major peaks related to polyisoprene structures but additionally shows a weak peak around 1558 cm^{-1} that is characteristic of the C=C stretching vibrations of

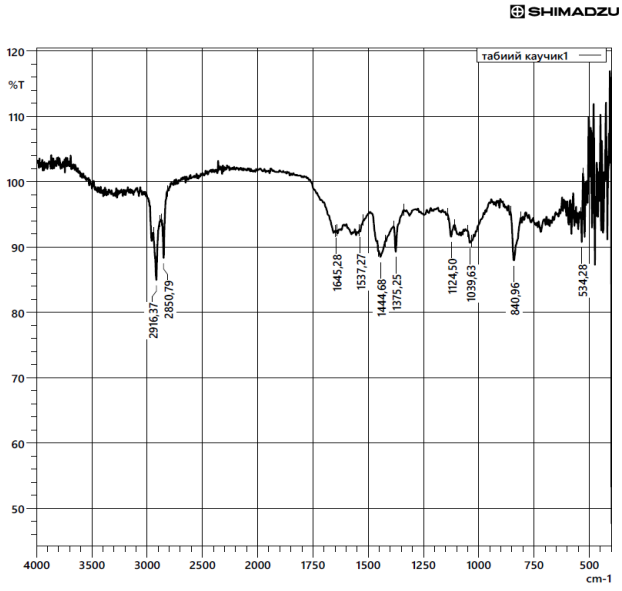
aromatic compounds with m-xylene domains. Analogously, the OG-T spectrum includes the peaks due to C=C stretching vibrations of aromatic compounds in the 1475-1427 cm^{-1} range, as well as aromatic out-of-plane C-H bending vibrations in the 910-830 cm^{-1} range. Such peaks indicate that the solvent environment with aromatic compounds had an impact on the network structure and was partly retained during polymerization. The low-intensity broad peak around 3313 cm^{-1} can possibly correspond to absorbed water molecules or polar groups present on the surface, yet its low intensity compared with other peaks means that the main matrix of the material is mainly nonpolar.



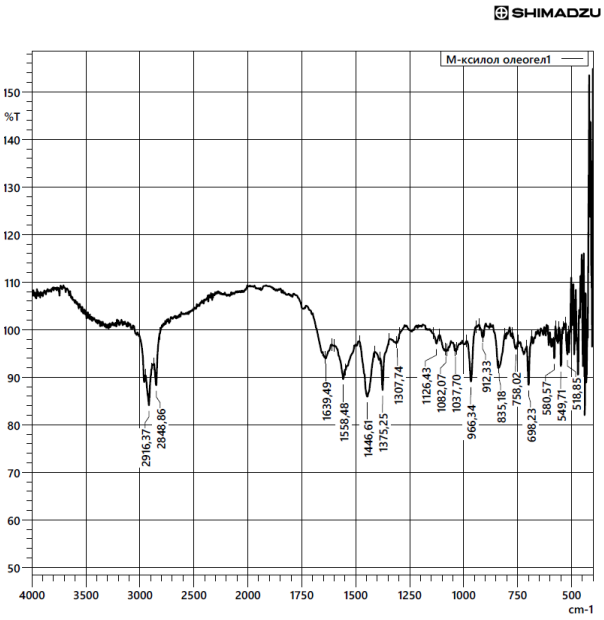
(a) SBR-1502



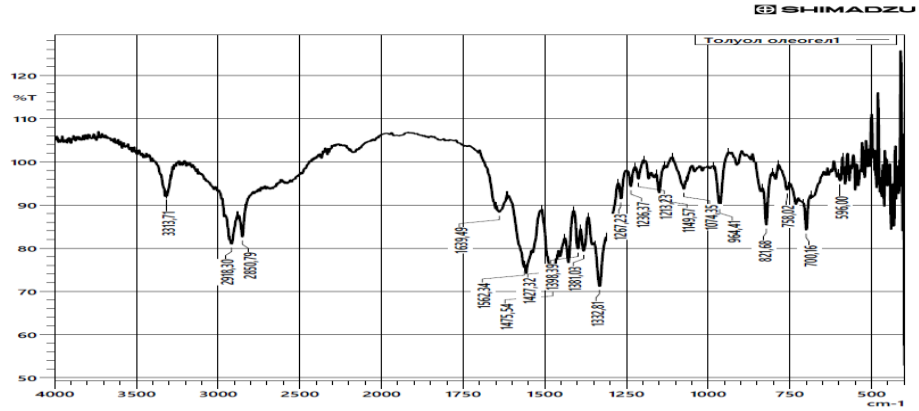
(b) SKI-3 NST



(c) SMR L



(d) OG-X



(e) OG-T

Figure 1. FTIR spectra of rubber precursors and oleogels obtained from: (a) SBR-1502, (b) SKI-3 NST isoprene rubber, (c) SMR L natural rubber, (d) OG-X, and (e) OG-T.

Mechanism of oil entrapment.

The suggested mechanism behind oil entrapment mainly relies on hydrophobic/oleophilic partitioning and entrapment. To begin with, it is worth mentioning that the surface energy of the hydrocarbon-based oleogel is better suited

for nonpolar substances than aqueous medium, which explains the preference of oil droplets toward the oleogel surface. Next, capillary forces help to pull the oil through the interconnected pores visible during SEM. In addition, polymer chain swelling and elasticity enable to entrain the oil within the three-dimensional network. Finally, aromatic groups in styrene-based domains as well as in solvents can provide additional interaction between aromatic compounds in oil contaminants.

These results, specifically the high percentage of carbon and low percentage of oxygen, indicate that hydrogen bonding and electrostatic forces are not the dominating forces behind the uptake. Oil adsorption will be governed by wetting, capillary action, expansion of the networks, and van der Waals forces. This explanation is consistent with the principle that governs superhydrophobicity and superooleophilic of porous materials used for sponges, aerogels, and rubber-based products.

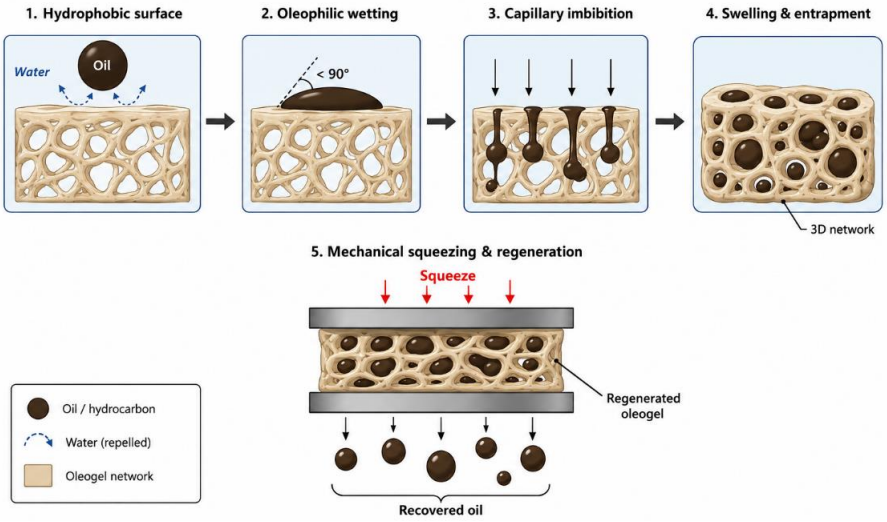


Figure 6. Proposed mechanism of oil adsorption by rubber-based oleogels: hydrophobic surface wetting, capillary imbibition, swelling of polymer network, entrapment of droplets, and physical removal.

Deployment cases for civil engineering applications.

The rubber oleogel compounds show promise for implementation in many different civil and environmental engineering applications. For use in industrial drain systems, such products would be useful in removing any remaining dispersed hydrocarbon particles from the water after gravitational or flotation treatment. For road drainage systems and vehicle servicing drains, the oleogels could be integrated into cartridge-style filters. At ports and ship repair yards, the more elastic OG-T varieties of oleogels could be used for creating floating barriers or mats. OG-X types of oleogels would likely find use at factory floor drainage and maintenance pits.

The choice between OG-T and OG-X will depend on how the material will be used. OG-T should be chosen when compression pads are required, floating sorbent layers are involved, and cyclic squeezing is employed. OG-X could possibly be chosen in cases when a hard network that does not deform under pressure is required, such as in cases when fixed cartridges, drainage layers, or packed bed filters will be used. Hydraulic performance should be tested before use in the field.

Conclusions

Oleogels containing rubber networks were synthesized based on a three-component mixture of SBR-1502, SKI-3 NST, and SMR L rubbers via benzoyl peroxide initiator and hexamethylenediamine in toluene and m-xylene solutions. The recipe for making oleogels containing rubbers was specified by mentioning the amounts of rubber, solvents, 10 wt.% initial solution, initiator, diamine, heating scheme, no subsequent washing, and drying in an oven at 40 °C for 24 hours. The FTIR spectra have proven that the oleogels synthesized maintain their hydrocarbon-rich polyisoprene/butadiene chain while including the presence of aromatic groups that suggest a network formation assisted by a solvent. A comprehensive batch adsorption experiment was devised, including model wastewater preparation, contact time, pH, temperature, concentration, dosages, kinetics and isotherms, regeneration, and statistical assessment. Initial data suggested rapid oleophilic wettability and oil uptake in 30-60 s at capacities estimated via gravimetry of 3.5-5.2 g/g for OG-T and 2.8-4.1 g/g for OG-X. Structure-property correlation data suggest that OG-T is the better material in terms of elasticity, compressibility, and regeneration capabilities, while OG-X might have advantages in applications requiring stiffer cartridges and packed bed sorption systems. This work establishes the foundation of the application of rubber oleogel technology in the realm of civil engineering in treating oily waste waters, industrial drainage, transport infrastructure storm water runoffs, and emergencies of oil spillage in an environmental cleanup context.

Foydalanilgan adabiyotlar

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