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OIL/WATER SEPARATION TECHNIQUES USING HYDROPHOBIZED/OLEOPHILIZED GRAINS: A REVIEW OF RECENT STUDIES

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ABSTRACT: Many domestic sources and industries, such as foods, textiles, metal industries, petrochemicals, and mining, produce massive volumes of oily wastewater all over the world. The insufficient treatment of oily wastewater in developing countries leads to serious environmental pollution and so on. In this review, we summarize many oil/water separation techniques to treat oily wastewater that have been developed such as flotation, chemical coagulation, biological treatment, filtration and membrane separation, and adsorption. Among them, oil/water separation techniques based on adsorption and filtration utilizing naturally hydrophilic sands and hydrophobized/oleophilized grains are worth attention due to their ease of application and cost effectiveness. In this review, we summarize recent studies on adsorption- and filtration-based oil/water separation techniques utilizing naturally hydrophilic sands and grains coated/modified with hydrophobic/oleophilic substances and compare the treatment performance, categorizing types of grains, coating substances, oil used, and testing methods. Prospects and challenges of oil/water separation studies are also given.

Keywords: Oily wastewater, Oil/water separation, Hydrophobized/oleophilized grains, Adsorption, Filtration

1. INTRODUCTION

The discharge of massive volumes of oily wastewater due to the rapid industrialization, unplanned urbanization, and agricultural development over the last few decades has had a serious impact on the environment and human health [1]. In particular, insufficient treatment and inadequate disposal of oily wastewater generated by human activities and industries such as petrochemical, food processing, textile, and metal, mining, biopharmaceuticals, and oil and gas refineries as well as oil-spill accidents are causing serious water pollution in developing countries. These negative phenomena are endangering aquatic life and significantly affect human health, nature, and the natural environment, which finally cause substantial economic losses [2].

In general, oil (as well as grease) in wastewater exists as various forms such as free and floating oil, dispersed oil (emulsified by a surfactant and/or mechanically), and dissolved oil. The concentrations of oil and grease in wastewater are highly dependent on the sources of the wastewater. Typical concentrations of oil and grease in untreated domestic wastewater range from 50 to 150 mg/L [3]–[4]. On the other hand, it is well known that industrial activities generate a wide range of oil and grease concentrations: e.g., oilfield-produced water contains 2–565 mg/L, wastewater generated by vegetable oil factories contains 480–7,782 mg/L, and wastewater/fluids from metalworking factories contain 20–200,000 mg/L [5].

Thus, treatment of oily wastewater is necessary all over the world, and permissible levels of oil and grease must be regulated before discharging the treated effluents into the environment. For example, the Water Pollution Prevention Act (1970) of Japan limits the maximum permissible levels of treated water discharge to 5 mg/L for mineral oil and 30 mg/L for animal and vegetable oils. In Vietnam, the permissible levels of animal and vegetable oils and grease in domestic wastewater range between 10–20 mg/L in QCVN 14:2008/BTNMT, depending on the destination of the discharge.

In recent years, many techniques for treatment of oily wastewater such as flotation, coagulation, chemical treatment, gravity separation, biological treatment, filtration and membrane separation, and adsorption have been developed [6, 7]. Among them, the oil/water separation techniques based on adsorption and filtration have been advanced by using various types of materials such as membrane, mesh, film, porous media, grains (powders), and so on. Not only natural hydrophilic/oleophobic (or natural hydrophobic/oleophilic) materials but also synthetic materials with hydrophobic/oleophilic substances for oil/water separation can be used to improve the surface hydrophobicity/oleophilicity [8–11]. Although various types of oil/water separation techniques have been reported, the further development of cost-effective and environmentally friendly (e.g., use of recyclable/reusable materials) methods is required, especially to treat large volumes of oily wastewater with high efficiency [12].

Among the oil/water separation techniques based on adsorption and filtration, the utilization of naturally hydrophilic sands and hydrophobized/oleophilized grains are of the most interest due to the ease of application and their cost effectiveness. In this study, we especially reviewed the recent progress in the utilization of naturally hydrophilic sands and hydrophobized/oleophilized grains for oil/water separation and compared the treatment performance, categorizing types of grains, coating substances, oil used, and testing methods.

2. METHODOLOGY

The data review was based on keywords such as oil/water separation, oily wastewater, hydrophobicity (oleophilicity), hydrophilicity (oleophobicity), hydrophobized/oleophilized grains, adsorption, and filtration. Tens of journal papers, books, and reports published in English after the year 2000 were searched in this study [e.g., Web of Science^{TM} (Clarivate Analytics)]. After checking the searched literature from the viewpoint of utilizations of hydrophobized/oleophilized grains for the oil/water separation, 16 papers were selected to summarize and compare the data.

3. RESULTS AND DISCUSSION

Table 1 summarizes the oil/water separation techniques utilizing naturally hydrophilic sands and hydrophobized/oleophilized grains, categorizing types of grains, coating substances, measuring the contact angle of water in air (CA_a) and the contact angle of oil in water (CA_o), oil used and concentration (or oil/water ratio), testing methods, and separation efficiency and/or separation capacity. The oil/water separation techniques are classified into two types: one is the adsorption-based technique, and the other is the filtration-based technique.

3.1 Types of Grains and Measured Contact Angles

For both adsorption-based and filtration-based oil/water separation techniques, sand materials were generally used for the oil/water separation techniques except for [23-26]. This is because sand materials are easily available, inexpensive, and nontoxic to environment [17]. Among the sand materials, quartz sand was commonly used for fabricating hydrophobicity/oleophilicity on the grain surface [14, 16, 21-22], and natural desert sand was used for the filtration-based oil/water separation due to its surface hydrophilicity/oleophobicity [18-20]. The tested grain sizes ranged <1 mm and were mostly around 0.2 mm, which is a typical boundary between fine and medium sand in the fields of geotechnical engineering and soil science.

CA_a is used to characterize the hydrophobicity/oleophilicity of a grain surface, and CA_o is used to characterize the hydrophilicity/oleophobicity of a grain surface. The relationship between measured CA_a/CA_o and the grain size is plotted in Fig. 1. The contact angles >150^o are identified as materials with a “superhydrophobic/superoleophilic” surface or a “superhydrophilic/ superoleophobic” surface [28]. The measured CA_a and CA_o were dependent on the tested oils, but most of the tested grain surfaces were >150^o, indicating that the tested grains were categorized as “superhydrophobic/superoleophilic” or “superhydrophilic/superoleophobic”. However, it has been reported that the grain size and geometry affected the water repellence characteristics of hydrophobized grains [29]. Thus, knowing the effects of grain size geometry on the surface is needed to select suitable materials for the oil/water separation techniques.

3.2 Used Oils for Oil/Water Separation Studies

As shown in Table 1, various types of oils such as gasoline, diesel, crude oil, vegetable oils (soybean cotton seed, rapeseed, and so on) including so-called volatile organic compounds (VOCs) such as petroleum ether, hexane, and chloroform were used depending on the purposes of the studies. In addition, both “oil in water” and “water in oil” were used, and both concentration (given in mg/L or mL/L in Table 1) and oil/water ratio (given in O/W in Table 1) were used in the oil/water separation studies.

3.3 Testing Methods and Separation Efficiency for Oil/Water Separation Studies

Basically, all reported studies on both adsorption- and filtration-based methods gave a good oil/water separation performance. The relationship between measured CA_a/CA_o and the separation efficiency and capacity are plotted in Fig. 2. For adsorption-based techniques using batch and mixing methods, the adsorption/adhesion capacities ranged between 110 and 500 mg/g. Atta [15] reported that the adsorption capacity of the crude oil was increased from 160 mg/g (uncoated silica particles) to 500 mg/g after coating the grain surface. Bigui [16] reported that the adsorption capacities of hydrophobized quartz sands increased 54% for the

Table 1 Oil/water separation techniques utilizing natural hydrophilic sands and hydrophobized/oleophilized grains.

Grains/sands (size in mm)	Coating substances	CA_a / CA_o (°)	Used oil*	Oil concentration or O/W	Method	Separation efficiency (in %) and/or separation capacity**	Ref.
Adsorption-based							
Silica particles (~0.4)	1,7-octadiene monomer	CA_a = 129-144	Motor oil, crude oil, kerosene	20 g/L (in water)	Batch (4 g solid to 80 mL oil)	> 99% 330-500 mg/g	[13]
Quartz sand (0.55-0.83)	ODTS, ZnO nanoparticles	CA_a = 150-154	Engine oil, rapeseed oil, and so on	N.C.	Mixing (10 g solid to 5 mL oil)	96.5-99.7% 180-380 mg/g	[14]
Sand (0.1-0.25)	TEOS, VTS	CA_a = 140-170	Crude oil	20 mL/L (in seawater)	Mixing (Sand : oil = 1:1, 1:2, 1:10)	80-92% (Coating substances: sand = 1:2)	[15]
Quartz sand (0.6-0.9)	Chitosan, SiO_2 nanoparticles	CA_o = 152	Engine oil, rapeseed oil	N.C.	Mixing (20 g solid to 100 mL oil)	99.9% ~ 280 mg/g (Engine oil) ~ 110 mg/g (Rapeseed oil)	[16]
Filtration-based							
Quartz sand (0.6-0.9)	Chitosan, SiO_2 nano-particles	CA_o = 152	Engine oil	O/W = 1:19 (in weight)	Gravity-driven (Porosity: 45 %)	~ 100%	[16]
Sand (0.1-0.5)	ODTS	CA_a = 155	Hexadecane, chloroform	O/W = 5:3 (in volume)	Gravity-driven with vacuum (Oil: chloroform)	High separation capacity remained even after 5 cycles of separation	[17]
Desert sand (0.13-0.27)	(Non-coating)	CA_o = 146-152	Decane, sesame oil, crude oil, diesel oil, etc.	N.C.	Gravity-driven (Oil: petroleumether)	High separation capacity depending on the thickness sand layer	[18]
Desert sand (> 0.05)	(Non-coating)	CA_o = 112*** (CA_a = 0)	Diesel, petroleumether, etc.	O/W = 50:1 (in volume)	Gravity-driven (Porosity: 79.6 %)	> 99.8% (Separation capacity was maintained even after 10 cycles of separation)	[19]
Desert sand (0.1-0.2)	Cu $(OH)_2$ nano-needles	CA_a = 158	1,2-dichloro-ethane	O/W = 2:1 (in volume)	Gravity-driven	Up to 99.5%	[20]

Table 1 continued

Quartz sand (0.55-0.83)	IDT, 3-MPS	N.D.	Gasoline engine oil	17.3 mg/L (in water)	Steady effluent flow rate	74-87% (in the first 9 hours)	[21]
Quartz sand (0.1-0.3)	HMDS (nano silica)	CA_o = 154	Xylene, chloroform, diesel oil	O/W = 1:1 (in weight)	Gravity-driven	97-98% (96% even after 50 cycles of separation)	[22]
Waste brick grains (0.1-0.4)	(Non-coating)	CA_o = 145.7 ± 2.8 (CA_a = 138.3 ± 3.4)	Soybean oil, hexane, etc.	N.C. (water-in-oil and oil-in-water emulsions)	Gravity-driven (Porosity: 47 %)	~ 98% (Separation capacity remained even after 10 cycles of separation)	[23]
Waste brick grains (0.05-0.4)	ZnO nanopillars	CA_0 = 152-153 (CA_a = 145-147)	Gasoline, Chlorobenzene, etc.	N.C. (water-in-oil and oil-in-water emulsions)	Gravity-driven (Porosity: 47 %)	95.5-98.5% (Separation capacity remained even after 10 cycles of separation)	[24]
Waste brick grains (0.05-0.4)	TiO_2	CA_0 = 148.6 ± 2.5 (CA_a = 143.8 ± 3.1)	Heavy oil	N.C.	Gravity-driven (Porosity: 47 %)	99.2%	[25]
Macro-porous ceramic materials (Al_2O_3)	PDMS	CA_a = 153.04 to 133.12	1,2-dichloro-benzene, Hexane, Isooctane, Dichloromethane, Petroleumether	O/W = 1:1 =10 ml:10 ml	Gravity-driven (Porosity: 65 %)	99.7 ± 0.92% (Separation capacity remained even after 10 times of separation)	[26]
Sand grains (<0.075)	PDMS	CA_a = 143 ± 3 to 152 ± 1)	Chloroform, Kerosene, etc.	O/W = 1:1=10 ml (Kerosene):10 ml (Water) O/W = 1:3=10 ml (Chloroform):30 ml (Water)	Gravity-driven	94-98% (Separation capacity remained even after 25 cycles of separation)	[27]

Note: *Used oil including volatile organic compounds (VOCs).**Capacity: Adsorbed amount of oil / grains (g/g) is given for the adsorption-based technique. Applicable range and observed performance of oil/water separation are given for the filtration-based technique. ***CA_o was read by the figure in the literature.

[Abbreviations] CA_a: Contact angle of water in air (°), CA_o: Contact angle of oil in water (°), HMDS: 1,1,1,3,3,3-hexamethyldisilazane, IDT: Isopropyl dioleic (dioctylphosphate) titanate, N.C.: Not controlled (used as is) or not clarified, N.D.: Not determined, ODTS: Octadecyltrichlorosilane, O/W: Oil/water ratio, PDMS: Dimethylpolysiloxane, TEOS: Tetraethoxysilane, VTS: Vinyltrimethoxysilane, 3-MPS: 3-methacryloxypropyltrimethoxysilane

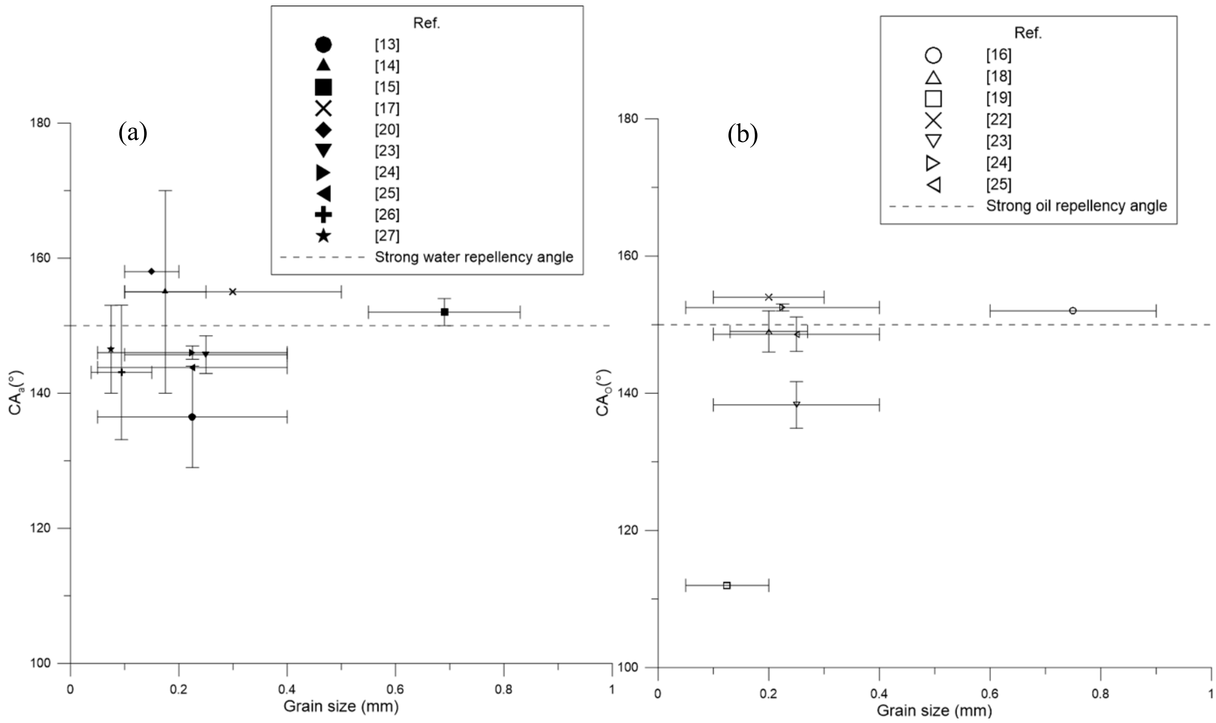

Fig. 1 Relationship between grain size and (a) Contact angle of water in air (CA_a), (b) Contact angle of oil in water (CA_o).

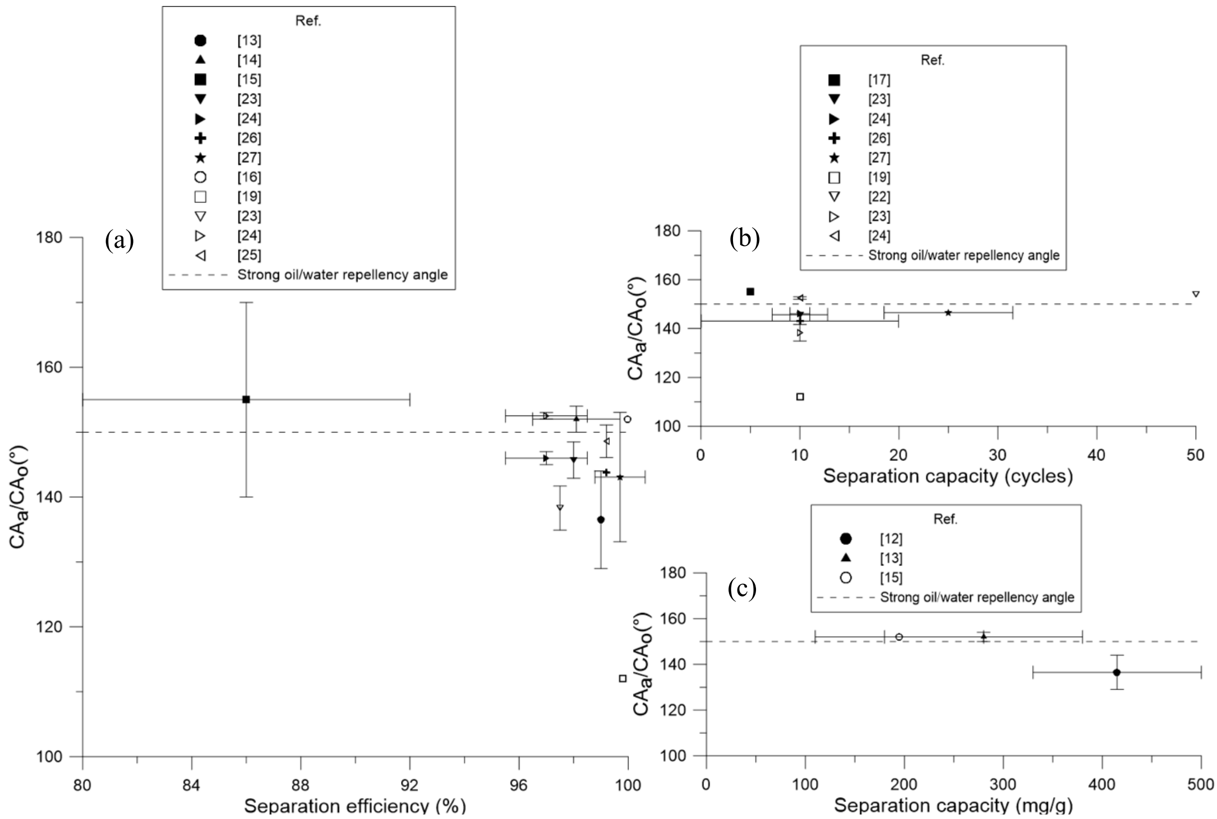

Fig. 2 Relationship between CA_a/ CA_o and (a) Separation efficiency (%), (b) Separation capacity (cycles), (c) Separation capacity (mg/g).

engine oil and 45% for the rapeseed oil compared to those of uncoated quartz sands. For filtration-based techniques, the gravity-driven method was commonly used to assess the separation efficiency and capacity. The separation capacity was validated based on the performance under repeated separation tests (i.e., cycles).

4. CONCLUSION AND PROSPECTS

In this paper, recent studies and progress on oil/water separation techniques based on adsorption and filtration utilizing natural hydrophilic sands and hydrophobized/oleophilized grains were reviewed. All reviewed methods performed well and gave high oil/water separation efficiency and capacity in the laboratory tests. Most of the studies used sands as tested grains. It is worth to note that Li [23] used grains from waste clay brick (originating from construction waste). In addition, Shi [30] utilized waste brick powders to coat a membrane for the oil/water separation. Junaid [31,32] assessed the water repellency of hydrophobized autoclaved aerated concrete grains that originated from scrap of construction materials. Use of these harmless waste materials and industrial by-products should be encouraged to utilize for the oil/water separation techniques from the viewpoints of reuse and recycle of waste, sustainability, and green development.

With regard to the practical applications of tested materials to the actual treatment of oily wastewater, not only assessment of the grain size and coating substances but also examination of suitable packing density (porosity) and flowing conditions of oily wastewater, such as line velocity, hydraulic retention time, and mass transport analysis under continuous flowing conditions, are important. Further studies and challenges are necessary to evaluate the performance of oil/water separation techniques utilizing naturally hydrophilic sands and hydrophobized/oleophilized grains, fully considering the packing density and flow conditions.

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