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Governing factors for motor oil removal from water with different sorption materials☆

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Abstract

This paper has been focused on the sorbent efficiency for motor oil removal from water. Two types of sorbents were investigated: organic and inorganic. Natural wool fibers (NWFs) and recycled-wool-based nonwoven material (RWNM)) were tested as organic type of sorbents. Sepiolite, bentonite and zeolite have been chosen as representative inorganic sorbents. Sorption was carried out in batch sorption system. Efficiency of oil removal was determined by measuring the oil concentration before and after the sorption process. Extractive-gravimetric method and refractive index determination have been applied as analytical methods for determination of oil concentration in water. Governing factors for sorbent efficiency were proposed, analysed and compared. It was concluded that sorption process is mostly affected by mass of sorbent, sorption time, temperature and pH value of water. NWFs, which were the most efficient sorbent showed maximal efficiency and maximal sorption capacity: 0.1 g of NWFs after 10 min at 20 °C and pH 8.00 sorbed 3.3 g of motor oil from 300 mL of water polluted with 4.5 g of motor oil. Maximal efficiency for all sorbents investigated was reached after 30 min of sorption processes, it was 95.0% for NWF, 43.0% for NRWM, 20.7% for sepiolite, 19.6% for bentonite and 21.2% for zeolite. Physical adsorption onto all sorbents is a favorable process (sorption efficiency decrease with increasing temperature) while sorption onto bentonite and zeolite is a result of both physical adsorption and chemisorption (sorption efficiency increase with increasing temperature, up to 80 °C).

Keywords: Motor oil; Wastewater; Sorption

1. Introduction

Environmental pollution of water by various types of oil has been, and continues to be a specific and serious problem. Investigations and development of new techniques are required, as well as improvements to known ones. In small plants and installations conventional purification processes are often combined with sorption processes. Sorption, as a stage of wastewater treatment could be efficient, not only for removal of oil, but also, for removal of all substances which have affinity toward physical adsorption or chemisorption. Sorbents usually contain various active groups, they are robust and can be a part of filtration medium. Multifunctional features of sorbents contribute synergetic effects during purification process. Sorbents, also, can be applied at any stage of oily wastewater treatment, without additional equipment in treatment plant.

Different types of sorbents, natural or synthetic, organic and inorganic, have been tested and investigated as possible sorbents for oil removal [1–9]. The major objective of this paper was to investigate a simple sorption procedure for motor oil removal from water. Two types of natural sorbents were used and investigated: organic and inorganic. Organic type of sorbents are based on wool: natural wool fibers (NWF) and recycled-wool-based nonwoven material (RNWM). Sepiolite, bentonite and zeolite have been chosen as representative inorganic sorbents (sepio-

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lite, from Serbia and bentonite, from FYRO Macedonia and zeolite, from Serbia) [10–15]. In order to enlarge the choice of material for oil removal by sorption process the aim of this study was to analyze, compare and extract the best features and efficiency of these sorbents. Motor oil was chosen as a model oil. Motor oil can be a serious environmental threat when disposed or reused improperly [16]. Sorption process was carried out in batch sorption system. The results of this study can be considered in making decision which type of sorbent can be efficiently applied for oil removal in a real wastewater treatment plant.

Wool fibers were chosen because of their high sorption capacity and biodegradability [4]. These features make these natural fibers very attractive as an alternative sorbent compared to synthetic fibers [8]. Other sorbents, bentonite, sepiolite and zeolite, were chosen because of their possible high efficiency, chemical and mechanical stability, high surface area and structural properties [12]. Sepiolite has the highest surface area of all the clay minerals [5,12]. High porosity and peculiar characteristics make sepiolite very efficient sorbents for removal of metal ions, dyes and various organic compounds from water [5,14]. Bentonite is a type of clay consisting dominantly of smectite minerals. Their sorption capabilities come from their high surface area and exchange capacities [11–13]. Zeolites are minerals that have a micro-porous structure. Zeolites are the aluminosilicate members of the family of microporous solids known as "molecular sieves". The term molecular sieve refers to a particular property of these materials, that is the ability to selectively sort molecules based primarily on a size exclusion process. This is due to a very regular pore structure of molecular dimensions. The maximum size of the molecular or ionic species that can enter the pores of a zeolite is controlled by the diameters of the tunnels. The pore openings for all rings of one size are not identical. Thus, some studies have been focused on removal of low-molecular-weight organic compounds from water by the use of zeolites [14–16].

2. Experimental

2.1. Chemicals

Chemicals used in experimental work were: sulphuric acid (H_2SO_4) (p.a., Aldrich); carbon tetrachloride (CCl_4) (p.a., Aldrich); *n*-hexane (C_6H_{14}) (p.a., Riedel de Haen); anhydrous sodium sulphate (Na_2SO_4) (p.a., Aldrich); methyl-*tert*-butyl-ether or MTBE ($C_5H_{12}O$) (Merck); hydrochloric acid (HCl) (1:1, Merck); nitric acid (HNO_3) (1:1, Merck).

2.2. Motor oil

Sorption experiments were conducted with motor oil (MO) that was dissolved in distilled water, and then used as a model oily wastewater. Investigated motor oil was characterized in the Laboratory of Belgrade Oil Refinery. The main physico-chemical characteristics of tested oil are presented in Table 1.

Table 1
Physico-chemical properties of MO (type: GALAX SUPER 3 SAE 30)

Parameter	Method	Value
The appearance	Visual	Clear
Water content and mechanical solids	ASTM D 2709	None
Density (kg/m^3)	ASTM D 1298	890
Viscosity (100 °C) (mm^2/s)	ASTM D 445	12.23
Viscosity index	ASTM D 2270	100
Flash point temperature (°C)	ASTM D 92	256
Cloudiness temperature (°C)	ASTM D 97	<−15
Ca content (wt.%)	AAS	0.21
Zn content (wt.%)	AAS	0.066
Total base number (TBN) (mg KOH/g)	ASTM D 2896	6.5
Foaming (mL/mL)	ASTM D 892	0/0; 0/0; 0/0;
Loss after evaporation after 24 h (%)	–	0.75
Loss after evaporation after 48 h (%)	–	0.84

2.3. Sorbents

Organic sorbents were natural wool fibers (NWF) and recycled-wool-based nonwoven material (RNWM). NWF originated from domestic sheep. The preparation of NWF and RNWM was already described in our previous paper [4]. Inorganic sorbents tested for oil removal were: sepiolite, from Kosovo, bentonite, from FYRO Macedonia and zeolite, from Serbia. Inorganic sorbent samples were mechanically activated in accordance with the following procedure. The sample was treated before using in the experiments as follows: natural samples were first cleaned mechanically from the visible impurities, and then were ground and sieved to obtain a 0.5 mm-size fraction. The suspension containing 10 g/L of inorganic sorbents was mechanically stirred for 24 h in distilled water, and after waiting a couple of minutes the supernatant suspension was filtered. Then, the solid sample was dried at 105 °C.

As a result of activation zeolite was in form of fine powder, bentonite had granulation of particles of 3 mm and sepiolite had granulation of particles ranging from 0.5 to 3 mm [8].

2.4. Analytical methods for determination of oil concentration

Direct analytical methods used for determination of oil concentration were: extractive-gravimetric method and instrumental methods (refractive index determination and FTIR spectrophotometry). Extractive-gravimetric method and refractive index determination were considered to be optimum methods because of the concentration of oil in samples. In addition, both methods provide superior precision and reliability [4,17–19].

Oil concentration (C_{oil}, mg/L), with extractive-gravimetric method was calculated according to the equation:

$$C_{\text{oil}} = \frac{(m_1 - m_2) \times 10^{-3}}{V_{\text{sample}}} \tag{1}$$

where m_1 is the mass of round flask with oil (g), m_2 the mass of dry and empty round flask (g) and V_{sample} is the volume of analyzed wastewater sample (L).

2.5. Experimental part

Oil sorption was investigated in the batch tank. Sorption in batch tank was conducted when sorbents were placed in Erlenmeyer flask (500 mL) with 300.0 mL of oily wastewater sample, with oil concentration of 4.50 g. Sample was then shaked in laboratory shaker for 30 min at 20 °C. Wool based sorbents were taken out of flask and drained for 1 min, while inorganic sorbents were removed after decantation. Sorbent efficiency was determined by the application of extractive-gravimetric method.

Sorption capacity for experiments in batch tank was determined according to the following equation:

$$q = \frac{C_i - C_f}{m} \quad (2)$$

where q is the sorption capacity (g/g), C_i the initial oil concentration (g), C_f the final oil concentration (g) and m is the mass of sorbent (g).

When organic sorbent was tested, the mass of sorbent varied from 0.050 to 0.175 g. When inorganic sorbent was tested the mass of sorbent varied from 2.50 to 12.50 g.

pH analysis were conducted within range 3.00–10.00. The pH adjustments were accomplished with 0.1 M HCl or 0.1 M NaOH.

For temperature influence on sorption efficiency the analysis were performed from 20 to 95 °C, for oily water solution.

When kinetics of the sorption was analysed 0.10 g of wool based sorbent (NWF or RNWM) or 5.00 g of each inorganic sorbent (S, B, or Z) were placed in Erlenmeyer flask (500 mL) with 300.0 mL of oily wastewater sample (prepared with by dissolving 4.50 g of MO in distilled water). pH of each prepared wastewater solution was 5.70. Sample and sorbent were then shaked in laboratory shaker for 5, 10, 30, 60, 120 and 240 min at 20 °C. Sorbent efficiency was determined by the application of refractometric index detection method for organic sorbents and by the application of extractive-gravimetric method for inorganic sorbents.

3. Results and discussion

3.1. Mass of sorbent

First of all the influence of sorbent mass on sorption efficiency during removal of motor oil from water was investigated. The results obtained with natural and inorganic sorbents are shown in Figs. 1 and 2.

It was concluded that oil sorption could be effectively accomplished by sorbents based on wool. Maximal sorption capacities of two type wool materials investigated were 33.0 g of oil/g sorbent for NWF and 18.5 g of oil/g sorbent, for RNWM.

All inorganic materials exhibited significantly lower sorption efficiency, almost the same size, mg/g. Maximal sorption capacities were 216 mg of oil/g of sepiolite, 174 mg of oil/g of bentonite and 170 mg of oil/g of zeolite.

The most efficient sorbent, for both inorganic and organic sorbents was NWF.

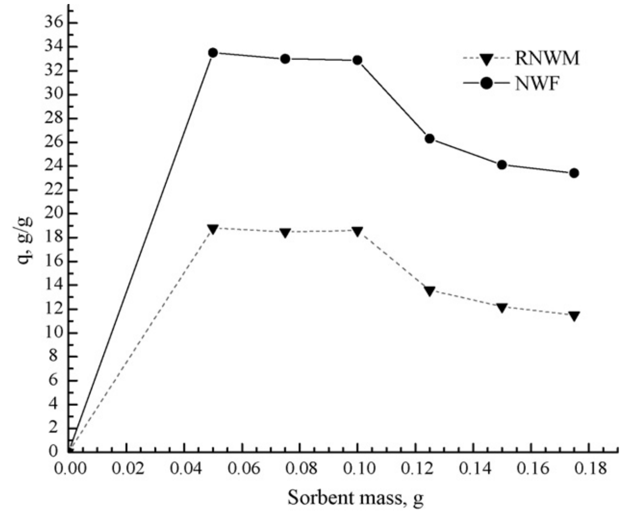

Fig. 1. Sorption efficiency for MO removal depending on mass of NWF and RNWM (τ = 30 min, m_{oil} = 4.5 g, $V_{oilywater}$ = 300 mL, pH 5.50 and t = 20 °C).

Figs. 1 and 2, also, illustrate the effect of saturation, after maximum sorption capacity was reached the efficiency of oil removal slowly decreased, since saturation of sorbent occured.

3.2. pH of wastewater solution

The effect of pH value of water polluted with MO on sorption process was studied and present in Table 2.

It was found that oil sorption on organic sorbents was highly affected by pH value which was expected, pH has strong influence to active group (–NH–, –CO–NH_2, –CO–NH–) on wool surface [8]. NWFs type was more sensitive to pH change then RNWM type. Optimal pH range for NWFs was from 8.00 to 10.00. Within this range NWFs were notable efficient up to 95%. For RNWM optimal pH was 3.00, when the efficiency of oil removal was 41%.

Concerning inorganic sorbents it was concluded that pH value has smaller influence to their sorption capacities compared with

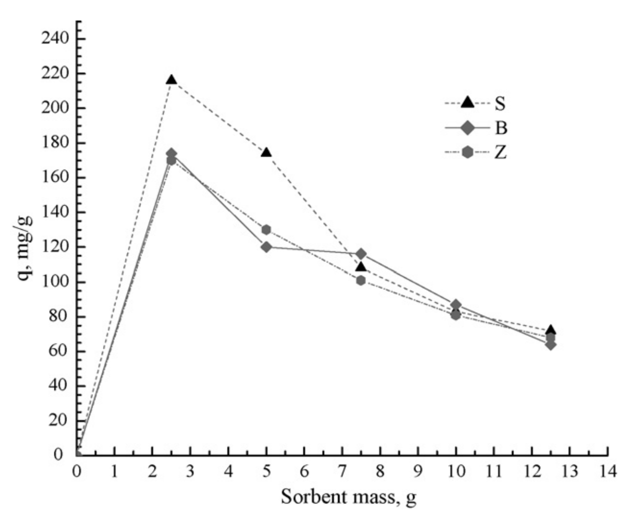

Fig. 2. Sorption efficiency for MO removal depending on mass of inorganic sorbents (τ = 30 min, m_{oil} = 4.5 g, $V_{oilywater}$ = 300 mL, pH 5.70 and t = 20 °C).

Table 2
The effect of pH on efficiency of oil removal with organic and inorganic sorbents ($m_{\text{organic sorbent}} = 0.1$ g, $m_{\text{inorganic sorbent}} = 5.0$ g, $m_{\text{oil}} = 4.5$ g, $V_{\text{oilywater}} = 300$ mL, $\tau = 30$ min and $t = 20$ °C)

pH	Efficiency of oil removal (%)				
	RNWM	NWF	S	B	Z
3.00	42.6	78.0	18.4	19.2	18.4
5.00	34.9	71.6	19.2	19.6	18.1
7.00	31.3	82.4	20.7	16.8	18.8
8.00	28.4	94.7	19.5	17.9	20.9
10.00	38.2	94.8	20.4	18.9	21.4

organic wool materials. It was observed that inorganic sorbents have different behaviour. Efficiency of oil removal with sepiolite and zeolite was increased with increasing pH value thou with bentonite efficiency decreased. Efficiency of sepiolite was increased up to 21%. The efficiency of sepiolite was high at two values of pH: neutral, and around 10.00. Bentonite exhibited its optimal removal (19%) in higher range of pH: from 3.00 to 5.00. When pH value was higher efficiency was lower (17%), and then again it raised with the increase of pH value. Maximum value for sorption capacity of bentonite was reached when pH value was 5.00 (174 mg oil/g sorbent). Zeolite exibited optimal removal (21%) when pH value was 10.00. Minimum efficiency zeolite exibited when pH value was 5.00 (18%).

When percentage of oil removal is considered the most efficient sorbent materials were NWF and zeolite, but when efficiency of oil removal is included NWF exibited the best total effect.

3.3. Temperature of oily wastewater solution

It was evident that there is a huge difference in the sorption capacities of materials concerning their organic and inorganic nature. Efficiency of organic (wool) sorbents to oil sorption from water is high, usually sorption of these type of materials are ascribed to physical adsorption. Efficiency of inorganic sorbent is relatively low, for these sorbents sorption is result of combined physical adsorption and chemisorption. Investigations of sorption on different temperatures can partially elucidate these aspects.

Temperature influence on MO removal by using organic sorbents is shown in Fig. 3. As it was concluded NWFs were more effective sorbent than RNWM at 20 °C, 33 and 19 g/g, respectively, but on higher temperature the efficiency decreased and equalized at lower value of 15 g/g. It is expected due to fibers degradation on higher temperature [20]. Since their structure and form is damaged, their functionality is also retained with the rise of temperature. When temperature interval was from 80 to 95 °C, the efficiency of both sorbent materials was very low. Their efficiency was only 2.5% and sorption capacity was around 1 g of oil/g sorbent material.

The effect of temperature on sorption of MO onto inorganic sorbents is shown in Fig. 4. Efficiency of sorption of MO on sepiolite decreases with increase of temperature. This could be

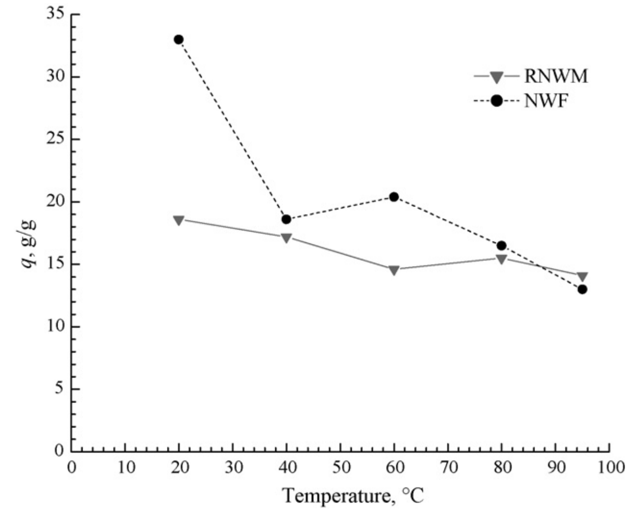

Fig. 3. Sorption capacity of NWF and RNWM vs. temperature for MO in water ($m_{\text{sorbent}} = 0.1$ g, $m_{\text{oil}} = 4.5$ g, $V_{\text{oilywater}} = 300$ mL, $\tau = 30$ min and pH 5.50).

ascribed to high contribution of physical adsorption as it was mentioned previously [20]. It should be emphasized that sorption is a dynamic process which is balance of sorption and desorption. Molecules of adsorbed material are in constant motion (Braunian motion) and when they are very close to the surface of sorbent they get attached and connected with the surface of sorbent. When temperature raises, the movement of molecules increase (motion is higher) and the interactions between sorbent and molecules are more intense. With higher temperature the possibility for molecules to attach to the sorbent surface is lower.

Sorption of oil on zeolite reached the percentage of oil removal of 18.4%, at 80 °C, which can be ascribed to the nature of zeolite that acts as molecular sieve [3]. Bentonite showed higher efficiency when temperature raise and it is probably due to higher contribution of chemisorption [10–13].

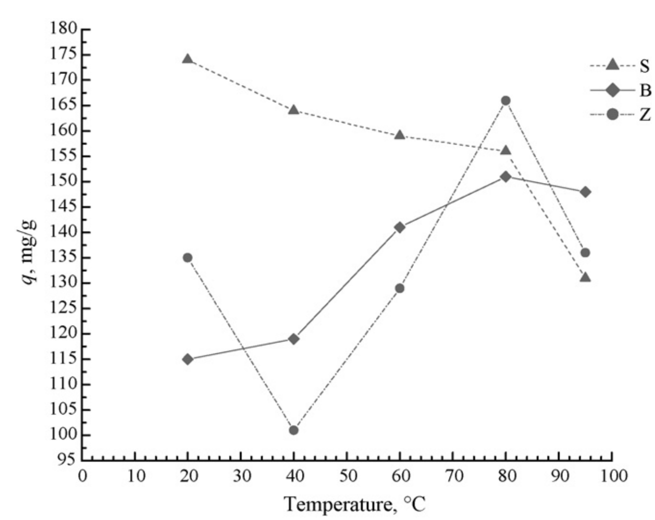

Fig. 4. Sorption capacity of inorganic sorbents vs. temperature of water polluted with MO ($m_{\text{sorbent}} = 5$ g, $m_{\text{oil}} = 4.5$ g, $V_{\text{oilywater}} = 300$ mL, $\tau = 30$ min and pH 5.70).

Table 3
The optimal experimental conditions and the effects of sorption with different sorbents analysed in this study ($m_{\text{motor oil}}$ = 4.5 g and $V_{\text{oilywastewater}}$ = 300 mL)

Sorption material	Optimal experminetal conditions				Efficiency of oil removal (%)	Sorption capacity (g oil/g sorbent)
	Sorption time (min)	Sorbent mass (g)	pH	Temperature (°C)		
NWF	10–30	0.03–0.04	8–10	20	73–95	33–43
RNWM	10–30	0.03–0.04	3–5	20	33–43	15–19
S	240	0.8–1.6	7–10	20	19.2–20.7	0.174–0.184
B	240	0.8–1.6	3–5	80	19.2–19.6	0.150–0.176
Z	240	0.8–1.6	8–10	60–80	18.4–21.4	0.166–0.192

3.4. Optimal experimental conditions for removal of motor oil in batch sorption system

After thorough investigation of sorption of MO with different natural sorbents (RNWM, NWF, S, B and Z) which included the function of the following parameters: mass of sorbent, time of sorption process, pH and temperature of water the optimal experimental conditions for motor oil removal are shown in Table 3.

In order to compare sorbents in Table 3 the sorption capacities and the efficiency for oil removal are considered.

NWFs, which were the most efficient sorbent showed maximal efficiency and maximal sorption capacity: 0.1 g of NWFs after 10 min at 20 °C and pH 8.00 sorbed 3.3 g of motor oil from 300 mL of water polluted with 4.5 g of motor oil. Maximal efficiency for all sorbents investigated was reached after 30 min of sorption processes, it was 95.0% for NWF, 43.0% for NRWM, 20.7% for sepiolite, 19.6% for bentonite and 21.2% for zeolite.

For wool based sorbents it was observed that after saturation point desorption process is prevailing and sorption capacity is lower. For inorganic sorbents this phenomena (desorption) was not observed. With the increase of sorption time the sorption capacity increases for all inorganic sorbents. After 240 min of sorption time for motor oil the sorption capacity was 216 mg of oil/g sorbent, 174 mg of oil/g sorbent and 170 mg of oil/g sorbent. When mass of sorbent was increased, in the batch sorption system, the higher efficiency of motor oil removal was achieved. The highest efficiency of oil removal was: 95% for NWF, 43% for NRWM, 20.7% for sepiolite, 19.6% for bentonite and 21.4% for zeolite. While sorption efficiency is higher, the sorption capacity in the batch tank is lower (opposite behaviour than in plug-flow system [4]). Unlike plug-flow systems, where concentration of oil is in constant contact with fresh surface of sorbent, the surface of sorbent in batch tank gets saturated as time of sorption is longer, and this, as expected, lowers sorption capacity.

4. Conclusion

The aim of this paper was to investigate a simple sorption procedure in batch sorption system for motor oil removal from water. In order to enlarge the choice of sorbents for oil removal a specific task of this study was to analyze, compare and extract the best features of two types of natural sorbents: organic and inorganic. Organic type of sorbents are based on wool: natural wool fibers (NWFs) and recycled-wool-based nonwoven material (RNWM). Sepiolite, bentonite and zeolite have been chosen as representative inorganic sorbents. It was obvious that there is a huge difference in the sorption capacities of materials concerning their organic and inorganic nature. Efficiency of organic (wool) sorbents to oil sorption from water is high due to physical adsorption. Efficiency of inorganic sorbent is relatively low, for these sorbents sorption is result of combined physical adsorption and chemisorption. Results obtained suggest that natural wool fibers are the most efficient for motor oil removal. NWFs, which were the most efficient sorbent showed maximal efficiency and maximal sorption capacity: 0.1 g of NWFs after 10 min at 20 °C and pH 8.00 sorbed 3.3 g of motor oil from 300 mL of water polluted with 4.5 g of motor oil. Maximal efficiency for all sorbents investigated was reached after 30 min of sorption processes, it was 95.0% for NWF, 43.0% for NRWM, 20.7% for sepiolite, 19.6% for bentonite and 21.2% for zeolite. Efficiency of sorption of MO on sepiolite decreases with increase of temperature. This could be ascribed to high contribution of physical adsorption. Sorption of oil on zeolite reached the percentage of oil removal of 18.4%, at 80 °C, which can be ascribed to the nature of zeolite that acts as molecular sieve. Bentonite showed higher efficiency when temperature raise and it is probably due to higher contribution of chemisorption.

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