JEE Journal of Ecological Engineering

Journal of Ecological Engineering 2024, 25(5), 208–218
https://doi.org/10.12911/22998993/185967
ISSN 2299–8993, License CC-BY 4.0

Received: 2024.02.24
Accepted: 2024.03.23
Published: 2024.04.01

Cost-Effective Remediation of Petroleum-Contaminated Waters Using Locally Sourced Wood Sawdust

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ABSTRACT

The increasing environmental pollution resulting from oil transportation, especially through pipelines such as the Baku-Supsa in Georgia, calls for the development of advanced wastewater purification technologies. This study investigates the use of wooden sawdust for the purification of oil-contaminated waters, aiming to utilize locally available residual natural materials for cost-effective environmental remediation. A comprehensive experimental methodology was adopted, involving thirteen types of plant-derived sawdust as sorbents to evaluate their oil sorption capacities under static and dynamic conditions. The effectiveness of these sorbents was assessed by their ability to lower the concentration of petroleum hydrocarbons in contaminated water, focusing specifically on the influence of sorbent particle size, contact duration, and the initial concentration of oil contaminants. The physicochemical characteristics of Azeri Light crude oil were detailed, and the sorption mechanism was scrutinized using gas-liquid chromatography to ascertain the fractional composition of the oil absorbed by the sawdust. Sawdust from Cryptomeria exhibited the highest oil sorption capacity, successfully absorbing 31.6 grams of oil per 100 milliliters of sorbent. Sawdust from Eucalyptus and Oak also displayed considerable sorption capabilities. The findings indicate that decreasing the particle size of the sawdust significantly enhances its capacity to sorb crude oil. Furthermore, steam-contact pre-treatment of the sawdust markedly increased its oil sorption capacity by 11% and tripled its efficacy in purifying oil-contaminated water. The results highlight the potential of employing locally sourced wooden sawdust, especially from Cryptomeria, as an efficient, sustainable, and cost-effective sorbent for cleaning oil-contaminated waters. The improved sorption capacity achieved through steam-contact pre-treatment presents a viable strategy for enhancing the performance of sawdust sorbents. This research contributes to the advancement of eco-friendly and economically feasible solutions for reducing water pollution caused by oil and its derivatives, emphasizing the critical role of sorbent selection and pre-treatment in refining purification processes.

Keywords: sorption, wooden sawdust, oil sorption capacities, petroleum hydrocarbons, wastewater.

INTRODUCTION

To mitigate environmental pollution, enhancing technological processes for wastewater purification is essential. Oil pipelines and terminals, notably the Baku-Supsa trunk oil pipeline in Georgia, pose significant risks of oil pollution. This pipeline, operational within Georgia, is pivotal in transporting Asian oil to European nations. Beyond the Supsa marine terminal, the region hosts several key terminals for oil and oil products, including the Black Sea Terminal in Kulevi, the Batumi Oil Terminal in Batumi, and the Channel Energy terminal in Poti. Process water, resulting from the draining or flushing of oil storage tanks, is laden with petroleum products, detergents, and oil sludge. This contaminated water is either processed through oil settling tanks for purification or, if untreated, discharged into sewers, rivers, or seas, escalating environmental risks.

The challenge in purifying water contaminated with oil lies in the emulsified state of oil products, creating a stable 'oil-in-water' emulsion. Mechanical, physicochemical, biological, and chemical methods are employed in the purification process, incorporating techniques such as coagulation (Puszkarewicz, 2008; Hassan et al., 2019; Zhao et al., 2021), flotation (Calcagnile et al., 2012; Zouboulis and Avranas, 2000), biological treatment (Yin et al., 2009; Gupta et al., 2011), membrane filtration (Janknecht et al., 2004; Tomaszewska et al., 2005), advanced oxidation processes (Santos et al., 2006) and integrated technologies (Benito et al., 2002; Beery et al., 2018). However, conventional approaches often have limitations. Mechanical containment and skimming are effective for large spills but may fail to capture dispersed oil fractions. Chemical dispersants can introduce harmful substances into the environment and pose risks to aquatic life. Biological treatments, such as biodegradation, may be slow and require optimal environmental conditions for effectiveness.

In response to the shortcomings of conventional methods, there is a growing need for economical and efficient remediation approaches. Biosorption, utilizing natural materials to adsorb pollutants, has emerged as a promising alternative. Lignocellulosic agro-forestry waste, such as sawdust, holds particular potential due to its abundance, low cost, and biodegradability. Before purification, the concentration of petroleum products in sediments is reduced to 50-100 mg/L. Sorption, recognized as the most effective physicochemical method post-initial purification stages (Sirotkina et al., 2005), employs various porous materials as sorbents. Key selection criteria for sorbents include non-toxicity, efficiency, affordability, reusability, and ease of disposal post-use (Radetić et al., 2003; Gołub abd Piekutin, 2018; Nechchadi et al., 2020;).

Percolation filtration through an adsorbent layer proves to be a simple yet effective method. Popular plant-derived sorbents include peat (Cojocaru et al., 2011), wood sawdust (Benyoucef et al.,2020), nanostructured cellulose (NSC) (Mahfoudhi and Boufi, 2017), cotton fibers (Deschamps et al., 2003; Lv et al., 2018) and straw (Sidiras et al., 2011). Among these, sawdusts from industrial and various agro-industrial wastes, such as diverse vegetable materials, offer the most cost-effective solutions. Notable examples include local fruit wastes (Alaa El-Din et al., 2018; Pires et al., 2021), maize stalk (Nwadiogbu et al., 2016), tea powder waste (Geetha et al., 2013), rice husk (Vlaev et al., 2011) and herbs (Zubaidi et al., 2015). The rationale for selecting sawdust as an oil sorbent lies in its porous structure and high surface area, which facilitate the adsorption of hydrophobic contaminants. Sawdust is readily available as a byproduct of wood processing industries and agricultural activities, making it an attractive option for large-scale remediation efforts.

The objectives of the current study are to evaluate the efficacy of sawdust-based biosorption for removing petroleum hydrocarbons from water and to assess its feasibility as a sustainable remediation strategy. Through laboratory experimentation and analysis, this research aims to contribute to the development of cost-effective and environmentally friendly solutions for oil pollution mitigation. The research aims to cost-effectively clean waters contaminated by oil and oil products. For this purpose, it is proposed that locally produced residual natural raw materials (wood waste, sawdust, and vegetable agro residues) be used as sorbents. To determine the optimal purification conditions, the impact of various factors, including the size of the sorbent particles, sorption time (contact time), and the initial concentration of oil products, on the efficacy of water purification was studied.

MATERIALS AND METHODS

Sorbents and oil properties

Thirteen types of plant sawdust samples were selected as research materials (Figure 1). These included pine (*Pinus*), hazel (*Corylus*), eucalyptus (*Eucalyptus*), hornbeam (*Carpinus*), plum (*Prunus domestica*), cryptomeria (*Cryptomeria japonica*), paulownia (*Paulownia*), oak (*Quercus*), alder (*Alnus*), mulberry (*Morus*), thuja (*Thuja standishii*), barberry (*Elaeagnus umbellata*), and cedar (*Cedrus*). The sawdust was prepared by natural drying method and was a mixture with a particle size of 1–4 mm. The cryptomeria sawdust was sieved into fractions of 0.5 mm, 1.0 mm, 2.0 mm, and 3.15 mm particle sizes. Raw sawdust samples were obtained from the city of Kobuleti from the industrial entrepreneur "Nodar" workshop, Georgia. The crude oil used in this study was Azeri light crude oil from Azerbaijan. This oil is offloaded at the Supsa Terminal at an annual volume exceeding 90,000 tonnes.

Figure 1. Sawdust from various plants

RESEARCH METHODS

The research was carried out following established standard methodologies. The standards applied included: ASTM D 5002, ASTM D 4052, ASTM D 4928, ГОСТ 6370-83, ASTM D 3230, IP 336, ASTM D 664, ASTM D 5853(A), ASTM D 86, ASTM D 5134 mod. For the research, the technical equipment utilized comprised the following: an X-ray fluorescence analyzer (RIGAKU NEX QC) for precise sulfur, a digital densitometer (Mettler Toledo DM-40), a coulometric Karl Fischer titrator (Mettler Toledo C-30) and an instrument for analyzing the fractional composition under atmospheric pressure (АРНС-1Э). A Thermo gas-liquid chromatography with a flame ionization detector, specifically the TRACE 1310 GC model, was used to analyze the composition of hydrocarbons. This setup was complemented by the Chrom-Card / PIANO automatic programming software and utilized a Supelco DH 100 quartz capillary column. The column measures 100 meters in length and 0.25 mm in inner diameter, with a methylsiloxane stationary phase. The employment of this particular column facilitates superior separation of hydrocarbons, extending to those in the C15 range. Chromatography conditions: TRACE 1310 GC model:

1. Analysis of detailed C5-15 hydrocarbons composition.
- carrier gas: helium, carrier pressure: 345 kPa; injector: split/splitless injector; split ratio: 200/1, inj. temp. set at 250 °C, FID det. temp. set at 250 °C, hydrogen flow rate: 35 mL/min; air flow rate: 350 mL/min; make-up gas flow rate: 20 mL/min; column: Supelco DH-100 100M, conditions: 35 °C for 13 minutes, rate 1: 10 °C/min to 45 °C hold 15 min, rate 2: 1.0 °C/min to 60 °C hold 15 min, rate 3: 2.0 °C/min to 180 °C hold 20 min. sample size: 0.5 ul.

2. Analysis of total hydrocarbons composition.
- carrier gas: helium, carrier 1 ml/min, injector: split/splitless injector; split ratio: 100/1, inj. temp. set at 280 °C, FID det. temp. set at 350 °C, hydrogen flow rate: 35 mL/min; air flow rate: 350 mL/min; make-up gas flow rate: 20 mL/min; column: DR-5MS-SA, 60 m, 0.25 mm, conditions: 30 °C for 14 minutes, rate 1: 5 °C/min to 300 °C hold 10 min, sample size: 1.0 ul.

Table 1. Physicochemical properties of Azeri Light Crude Oil

Test	Methods	Unit	Results
Density at 20 °C	ASTM D 5002	kg/L	0.8453
Density at 15 °C	ASTM D 5002	kg/L	0.8488
Water content	ASTM D 4928	Mass %	0.16
Sediments	ASTM D 473	Mass %	0.01
Salts content	ASTM D 3230	lb/1000bbl(PTB)	2
Salts content	ASTM D 3230	mg/kg	6.7
Sulphur content	IP 336	Mass %	0.16
Acid number	ASTM D 664	mg KOH/g	0.45
Pour point	ASTM D 5853(A)	°C	Minus 3
Distillation	ASTM D 86		
IBP		°C	56.0
100°		%	4.0
120°		%	8.0
150°		%	14.0
160°		%	16.5
180°		%	20.0
200°		%	24.5
220°		%	28.5
240°		%	32.0
260°		%	37.0
280°		%	42.5
300°		%	50.0
10%		°C	137.0
20%		°C	180.0
30%		°C	234.5
40%		°C	274.0
Recovery at 300 °C		%	51.5

EXPERIMENTAL PART AND DISCUSSION

Sorption process

In the preliminary phase of the study, sawdust from 13 different plant species (both broad-leaved and coniferous) was tested to determine which had the best oil sorption capacity under dynamic conditions. One hundred milliliters of each sawdust type was collected and ground to a particle size of 1–4 mm. The sawdust samples were loaded into chromatographic columns with dimensions of 250 mm height × 50 mm diameter. Oil was then passed through each column. The quantity of oil absorbed by the sawdust was quantified by measuring the mass difference before and after the experiment. This approach facilitated the determination of the sorption capacity of sawdust from various plants, expressed in both mass-to-mass percentage (m/m%) and volume-to-volume percentage (v/v%). Additionally, the mass, volume, and density of the sawdust loaded into the columns are detailed in Table 2. Of the plant species investigated, Cryptomeria sawdust exhibited the highest oil sorption capacity, absorbing 31.6 grams of oil per 100 milliliters of sorbent. Notably, sawdust from Eucalyptus and Oak also demonstrated commendable performance, showing equal sorption capacities of 26.6 g/100 mL and 26.0 g/100 mL, respectively. In contrast, Mulberry sawdust presented the lowest sorption capacity in this study, with an absorption rate of 17.0 grams per 100 milliliters. Research has demonstrated that oil sorption capacity does not correlate with sawdust density. Rather, it appears to be determined by other structural factors within the plant cell walls.

Table 2. Oil sorption capacity of sawdust from different plant species

No.	Sample name	Volume, ml	Weight, g	Density, g/ml	Sorption capacity, g/100 ml	Sorption capacity, g/g	Sorption capacity, m/m%	Sorption capacity, v/v %
1	Pine (*Pinus*)	100.00	17.12	0.1712	23.7	1.4	138.7	28.1
2	Hazel (*Corylus*)	100.00	17.28	0.1728	19.9	1.2	115.4	23.6
3	Eucalyptus (*Eucalyptus)*	100.00	14.61	0.1461	26.6	1.8	182.3	31.5
4	Hornbeam (*Carpinus*)	100.00	14.51	0.1451	23.7	1.6	163.2	28.0
5	Plum (*Prunus domestica*)	100.00	17.42	0.1742	24.3	1.4	139.4	28.7
6	Cryptomeria (*Cryptomeria japanica*)	100.00	12.53	0.1253	31.6	2.5	252.2	37.4
7	Paulownia (*Paulownia*)	100.00	9.85	0.0985	21.2	2.2	215.5	25.1
8	Oak (*Quercus*)	100.00	23.78	0.2378	26.0	1.1	109.4	30.8
9	Alder (*Alnus*)	100.00	11.91	0.1191	22.9	1.9	192.4	27.1
10	Morus (*Morus*)	100.00	10.19	0.1019	17.0	1.7	166.7	20.1
11	Thuja standishii (*Thuja standishii*)	100.00	9.95	0.0995	18.6	1.9	186.7	22.0
12	Elaeagnus umbellata (*Elaeagnus umbellata*)	100.00	11.11	0.1111	18.1	1.6	163.2	21.5
13	Cedrus (*Cedrus*)	100.00	11.85	0.1185	21.6	1.8	182.0	25.5

Effect of sawdust particle size on oil sorption capacity

To determine the influence of sawdust particle size on oil sorption capacity, sawdust samples were sieved using sieves with mesh sizes of 0.5 mm, 1 mm, 2 mm, and 3.15 mm. For this study, Cryptomeria and pine sawdust samples (volume 100 ml), spanning particle sizes of 0.5 mm, 1 mm, 2 mm, and 3.15 mm, were selected to evaluate their crude oil sorption capacities. The research findings indicate a direct correlation between particle size and sorption capacity: as the particle size decreases, the sorption capacity for crude oil correspondingly increases. The results of this study are detailed in Table 4 for pine sawdust and in Table 5 for Cryptomeria sawdust.

Purification of water containing crude oil using the sorption method

Sawdust derived from different plant species contains various resinous and organic compounds. For a reliable study of the sorption properties of sawdust, it is necessary to first remove phenolic, tanning and other substances that are contained in sawdust and can pass into the aquatic environment. This may lead to erroneous results. For pre-cleaning were used the green solvents such as water and ethanol (Kvasenkov et al., 1993; Santos et al., 2022**)**

Procedure was implemented as follows: 100 mL of the sorbent was washed with 200 mL of 96% ethanol, followed by an equivalent volume of distilled water. This washing process was conducted at a flow rate of 3.2–5.0 mL/min.

Table 3. Effect of pine sawdust particle size on oil sorption capacity

No.	Particle size, mm	Weight, g	Density, g/ml	Sorption capacity, g/100 ml	Sorption capacity, g/g	Sorption capacity, m/m%	Sorption capacity, v/v %	Sorption capacity, g/100 ml
1	0.5	25.21	0.2521	46.7	55.2	1.9	185.2	55.2
2	1.0	23.52	0.2352	36.2	42.0	1.5	153.9	42.0
3	2.0	18.12	0.1812	23.3	27.0	1.3	128.4	27.0
4	3.15	15.52	0.1552	19.8	23.0	1.3	127.7	23.0

Table 4. Effect of cryptomeria sawdust particle size on oil sorption capacity

No.	Particle size, mm	Weight, g	Density, g/ml	Sorption capacity, g/100 ml	Sorption capacity, g/g	Sorption capacity, m/m%	Sorption capacity, v/v %	Sorption capacity, g/100 ml
1	0.5	18.45	0.1845	56.20	66.49	3.0	304.6	66.5
2	1.0	16.14	0.1614	48.20	57.02	2.6	257.4	48.2
3	2.0	13.12	0.1312	31.00	36.67	2.0	203.7	31.0
4	3.15	11.05	0.1105	26.40	31.23	2.1	205.9	26.4

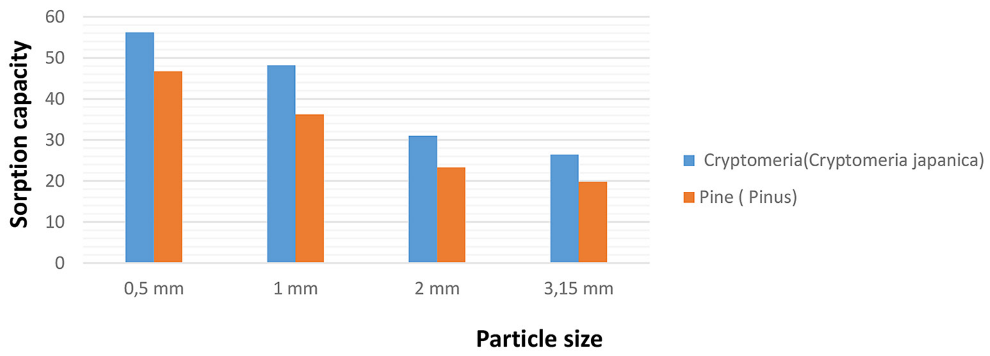

Figure 2. Correlation between the sorption capacity of wood sawdust and the particle size of samples

Table 5. Analysis of C5-C15 hydrocarbon contaminated in water before and after sorption

Research solution	Analysis method	Unit	Content of oil components
Initial solution	GC	mg/L	29.25
After sorption in sawdust	GC	mg/L	11.55

An experimental simulation was conducted to determine the effectiveness of cleaned sawdust in treating water contaminated with crude oil. To create a realistic simulation of polluted water, 100 ml of Azer light crude oil was mixed with 900 ml of water. This mixture produced an oil-water emulsion with an oil mass fraction of 8.57%, resulting in a total mass of 984.5 g (84.5 + 901 g) when accounting for both oil and water components. The emulsion was prepared with a 1:9 oil-to-water ratio, aiming to closely mimic the conditions of water affected by crude oil contamination. Cryptomeria sawdust was selected as the sorbent due to its superior sorption capacity compared to other plants evaluated in the study.

To quantify the oil components removed from the water, an extraction process utilizing chromatographically pure n-heptane was employed. This procedure entailed extracting oil components from both the original oil-water mixture and the filtrate that had been treated with the sorbent. Subsequently, the extracted samples underwent analysis with a gas-liquid chromatograph (Thermo TRACE 1310). The results indicated a significant decrease in the concentration of oil components in the water, with a reduction of 39.49% following the sawdust treatment. Maximum capacity absorption of C5-C15 Hydrocarbon in sawdust is 300 ml. Detailed outcomes of this research are presented in Table 6.

Analysis of c5-c15 hydrocarbon composition in oil-contaminated water before and after sorption

The objective of this study was twofold: firstly, to evaluate the purification efficiency of the chosen sawdust sorbent in treating water contaminated with crude oil, and secondly, to identify which hydrocarbon fractions from the crude oil were transferred into the water and which were effectively absorbed by the sorbent.

The analysis involved the examination of both the water containing crude oil and the filtrate resulting from its passage through the sorbent. The composition of individual hydrocarbons within

Table 6. C5-C15 hydrocarbon composition in oil-contaminated water before and after sorption

Time	Component	Initial water with crude oil, mg/L	Water after sorption, mg/L
13.00	Cyclopentane	0.50	0.50
13.30	2-Methylpentane	0.25	0.10
14.15	3-Methylpentane	0.25	0.50
15.25	n-Hexane	0.25	0.10
17.36	Methylcyclopentane	0.25	0.10
18.19	2.2.3-Trimethylbutane	0.50	0.50
19.52	Benzene	0.25	0.15
31.21	Ethylcyclopentane	5.00	2.00
35.28	Toluene	1.00	1.00
55.90	Ethylbenzene	13.00	4.50
58.36	m-Xylene	1.00	0.50
58.68	p-Xylene	3.50	1.50
64.27	o-Xylene	1.00	0.05

the C5-C15 fraction that had been transferred to the water was determined using gas-liquid chromatography. The findings from this analysis are detailed in Table 7. Based on the data, it is evident that compounds such as Cyclopentane, 2,2,3-trimethylbutane, and Toluene exhibit low sorption onto sawdust in oil-water systems.

Sorption of crude oil compounds on steam-treated sawdust

Due to the presence of various resinous and organic compounds in wood sawdust, some of these compounds are released into water upon dissolution of the sawdust. Hence, pre-cleaning (activation) of the sawdust intended for sorption was conducted. To achieve this, a specialized device was developed to clean (activate) the sawdust using high-pressure and high-temperature steam.

Following the pre-cleaning process, both pure oil and oil-saturated water were passed through the treated sorbent (sawdust). For the experiments, a simulated polluted water sample was prepared by mixing 25 ml of oil (specifically, Azer light crude oil of medium density) with 975 ml of water. This mixture resulted in water saturated with oil, containing 123 mg/l of petroleum products (Figure 4). Cryptomeria sawdust was selected as the sorbent due to its superior sorption capacity compared to the other investigated sawdust types.

To quantify the retention of oil components from water, both the sorbent and the oil-contaminated water were extracted using n-pentane

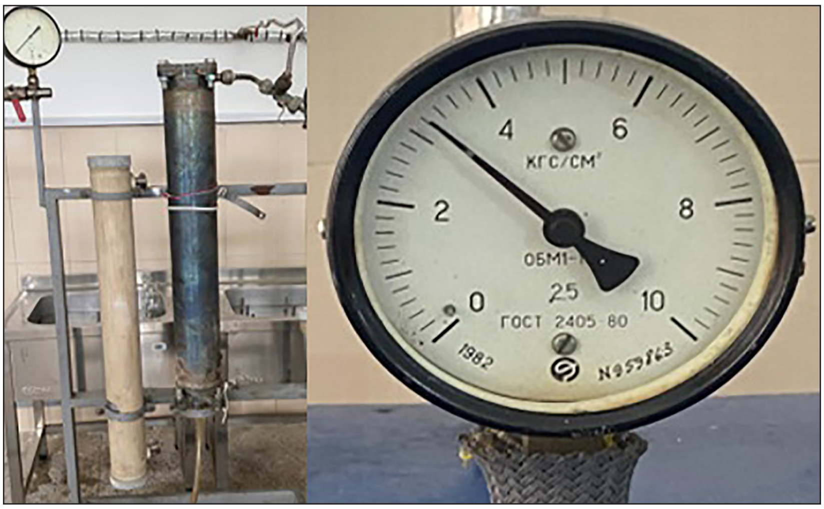

Figure 3. Column for cleaning sawdust with steam, T = 140–160 °C, P = 2.5–3.2 atm

Table 7. Reduction in petroleum hydrocarbon content following treatment with activated sawdust

Research solution	Volume, ml	Unit	Content of crude oil compounds, GC method
Initial solution		mg/L	123
After sorption in sawdust	50	mg/L	0.00
After sorption in sawdust	100	mg/L	1.15
After sorption in sawdust	200	mg/L	16.07
After sorption in sawdust	500	mg/L	36.43

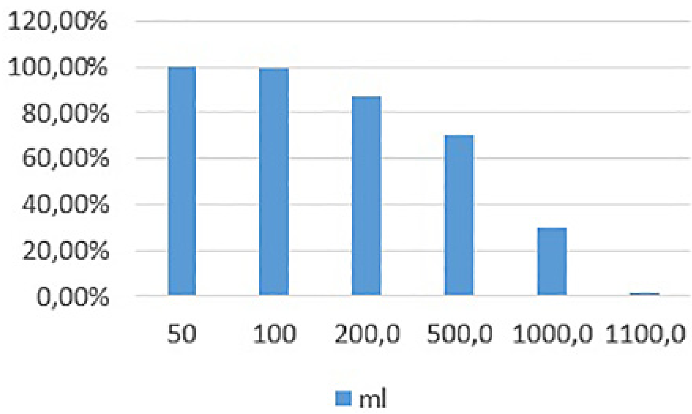

Figure 4. Efficiency of the sorbent

Table 8. Dependence of the efficiency of the sorbent on the volume of contaminated water

Research solution	Volume, ml	Efficiency of the sorbent
After sorbtion in sawdust	50	100.0%
After sorbtion in sawdust	100	99.1%
After sorbtion in sawdust	200	86.9%
After sorbtion in sawdust	500	70.4%
After sorbtion in sawdust	1000	30.1%
After sorbtion in sawdust	1100	1.6%

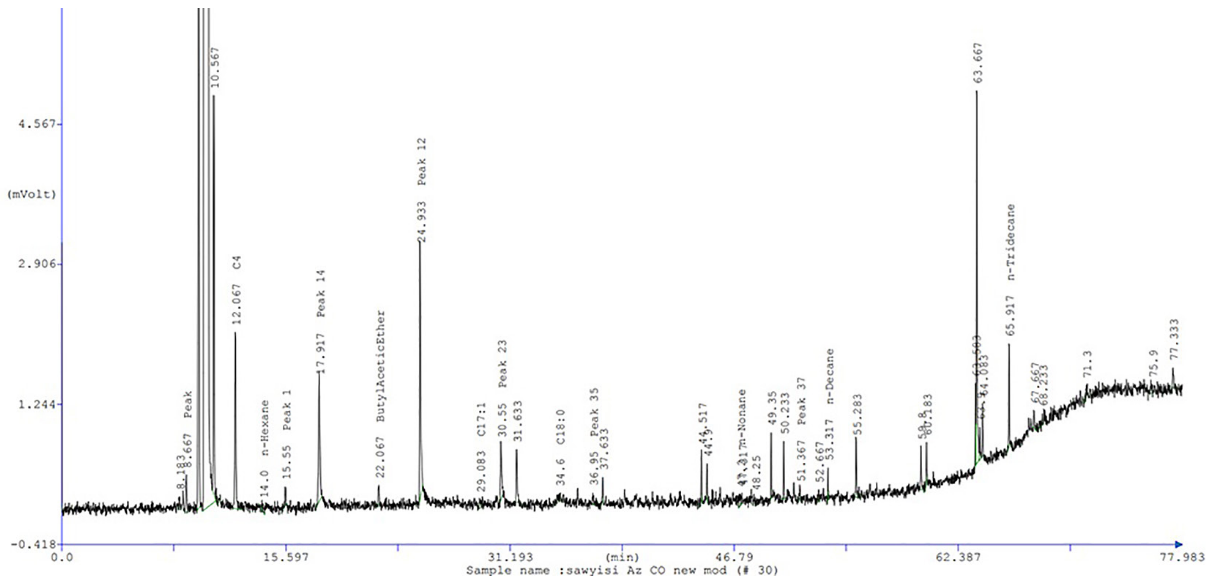

Figure 5. Before treatment: the content of petroleum hydrocarbons was measured at 123 mg/L

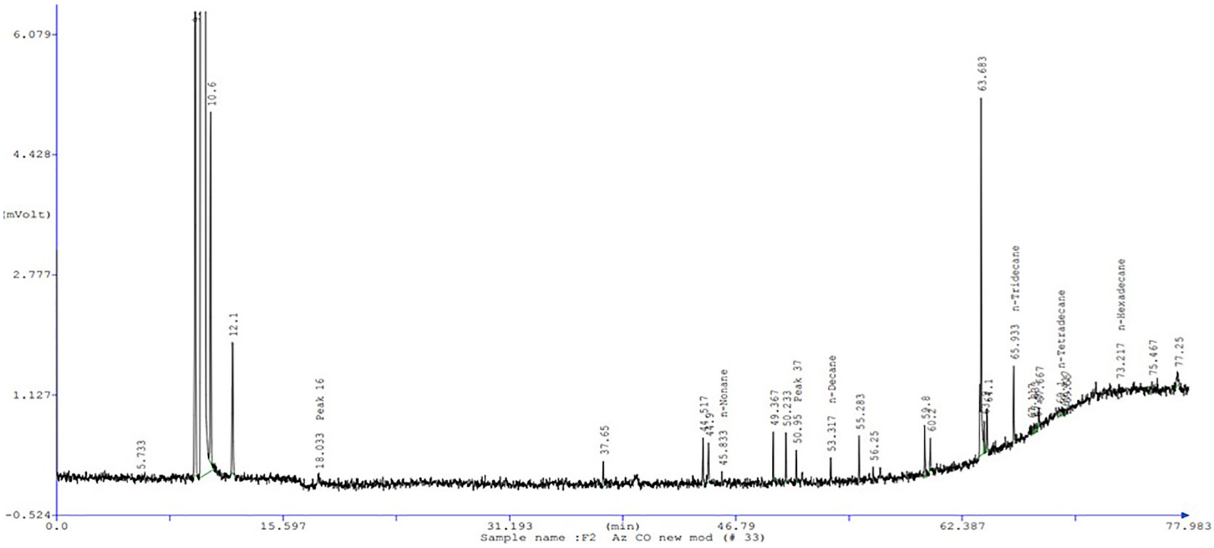

Figure 6. After treatment: after passing through the sorbent (content of petroleum hydrocarbons 16.07 mg/l)

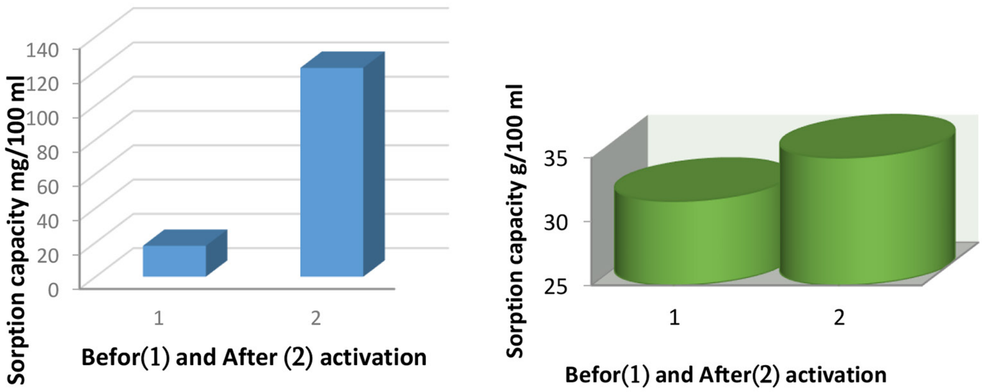

Figure 7. (a) Increased oil absorption after activation of sawdust, (b) an increase in the sorption activity of the crude oil in water after activation of sawdust

(chromatographically pure). The concentration of petroleum hydrocarbons was then measured using gas chromatography. The experimental results demonstrate a significant reduction in the concentration of oil components in water after treatment with sawdust. Specifically, the oil content decreased to 0 mg/L after treating 50 mL of contaminated water. Upon treating 100 mL, the oil concentration was reduced to 1.15 mg/L. For a volume of 200 mL, the concentration dropped to 16.07 mg/L (Figure 5); after treating 500 mL, it further decreased to 36.43 mg/L. The process of purifying petroleum water was carried out at 25 °C in vertical columns at a flow rate of 3–5 ml/min. The results of the study are presented in Table 9.

The maximum amount of water passed through was identified, after which the sorption capacity of activated sawdust is saturated, which corresponds to 1100 ml per 100 ml of sorbent. Steam-contact treatment of the sawdust increased the oil sorption capacity by 11%. For oily water, this pre-treatment improved the sawdust's efficiency at 18 mg/100 ml to 121 mg/100 ml. Which is 6.7 times more after activation

CONCLUSIONS

The study investigated the sorption of petroleum hydrocarbons by sawdust from thirteen different plant species under both static and dynamic conditions. The sorption capacity, measured in volume/volume percentage (v/v%), was determined for each type of plant sawdust. Among the tested materials, Cryptomeria sawdust exhibited the highest sorption capacity, absorbing 31.6 grams of oil per 100 milliliters of sorbent. Eucalyptus and oak sawdust also showed significant sorption capacities, with 26.6 g/100 ml and 26.0 g/100 ml, respectively. Using pre-treated Cryptomeria sawdust as a natural sorbent, the purification of simulated oil-contaminated water was achieved, and the reduction in petroleum hydrocarbon content was quantified post-sorption through chromatographic analysis.

Poor sorption from water to sawdust was observed for specific oil components including cyclopentane, 2,2,3-trimethylbutane, and toluene. Steam-contact treatment of the sawdust increased the oil sorption capacity by 11%. For oily water, this pre-treatment improved the sawdust's efficiency at 18 mg/100 ml to 121 mg/100 ml, which is 6.7 times more after activation.

Acknowledgements

This research was financed by the Shota Rustaveli National Science Foundation of Georgia under Project FR-22-2857 „Development of Ecologically Appropriate, Waste-Free Technological Processes for the Treatment of Oil-Polluted Water".

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