

## Performance evaluation of *Solanum incanum* leaves as a biodegradable adsorbent for oil-spill cleanup in seawater

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#### ABSTRACT

Leaves of *Solanum incanum* were used as oil adsorbent biomass in oil sorption from seawater. The capability of biomass to eliminate oil from seawater was related to its surface structure. The parameters of spilled oil ratio, sorption time, biomass dose, and the temperature of the oil/ seawater system were studied. Maximum oil sorption capacity (11.54 g/g) was obtained at 4 min and room temperature (28°C). The best fitting of Redlich–Peterson and Langmuir isotherm corroborates the homogeneous monolayer oil sorption process onto sorption sites of biomass rather than heterogeneous multilayer oil sorption. The pseudo-first-order kinetic model ( $R^2$ : 0.97941 and  $\chi^2$ : 0.27082) provided good fitting compared to the pseudo-second-order ( $R^2$ : 0.95173 and  $\chi^2$ : 0.63476) and intraparticle diffusion models ( $R^2$ : 0.76666 and  $\chi^2$ : 3.06843) indicating the physical nature of oil sorption onto biomass. Moreover, the reusability results demonstrate acceptable sorption effectiveness of *Solanum* leaves upto three sorption cycles.

Keywords: Sorption; Oil spill; Solanum incanum; Kinetic; Isotherm.

#### 1. Introduction

Oil is the most widely used fossil fuel and is a natural supplier of energy in the world along with natural gas. Oil has played a major role in determining the economic structure of the world [1,2]. The recent increase in oil spill pollution of seawater has alarming effects on the environment and people's health [3,4]. Oil spill pollution results in a significant decrease in the amount and quality of food produced by the sea, particularly the food protein required to feed the growing population and the marine wealth [5]. Moreover, oil spills reaching the shores can be damaging to the tourism industry due to the distortion of the environment, and hence, there should be measures taken, treatments ensured, standards set, and legislations enacted to reduce this type of pollution [6,7]. Oil pollution can be caused by the following: explosions and sinking of oil tankers, offshore oil wells explosions, accidents of defects in the charging and discharging currencies, cargo ships, tankers, and oil platforms' waste, petrochemical plants situated on the seashore, the natural leakage of oil from the sea and ocean floor, and attacks on oil installations and oil tankers during war operations [8]. Oil spill on surfaces leads to the release of several gases, including methane, ethane, propane, and butane, as well as polycyclic aromatic gases that are considered among the most toxic gases and cause respiratory cancers [9]. Leaked oil also has detrimental effects on factories and oil refineries since it can lead to fires or explosions and be a threat to desalination plants since drinking water can

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mix with toxic hydrocarbons, which may, in turn, result in the closing of these stations [10]. Owing to the technological development in the oil industry, several treatment methods have been established for oil spills removal; these methods eliminate the polluting effects and facilitate the whole process [11]. Different methods have been devised to treat oil spills, such as biological treatment through the addition of nutrients containing nitrogen and phosphorous and acceleration of the natural biological decomposition of oil by bacteria [12]. Another method is to wash by the effect of breaking waves, which speeds up the natural decomposition of oil. Besides, the skimming that collects spilled oil has been improved [13]. Besides enabling the recovery of oil through mechanical methods, the process of removing leaked oil using absorbent materials is important and cheap [14]. Many materials have shown a high ability to absorb oil from seawater from these materials agricultural wastes which are characterized by their low cost, availability, high absorbency, and natural decomposition [11]. However, one of the disadvantages that limit the use of the absorption process is its massive production of solid waste, but this point has been overcome by employing it as fuel in many processes, such as the production of steam in steam boilers. Considerable natural biodegradation biomass is employed in the oil sorption system, including sugarcane bagasse [15], vegetable fibers [16], barley straw [17], raw luffa [18], kapok [19], Ceiba pentandra [20], and wheat straw [21]. A new biodegradable adsorbent material was utilized to sorb oil from seawater in this study. Solanum incanum is a plant that grows naturally in southern Saudi Arabia and is utilized in leather tanning and medicinal compounds. Its leaves are used as new adsorbent biomass to eliminate oil from seawater in a batch system. Operating parameters (spilled oil ratio, sorption time, biomass dose, and the temperature of oil/ seawater system), kinetics, and sorption isotherms were studied. Also, it was investigated whether these leaves could be reused.

#### 2. Materials and methods

#### 2.1. Preparation of biomass

*S. incanum* leaves were obtained from the local market. They were washed with distilled water several times to eliminate foreign matters, dried at 85°C for 24 h, and then ground into 75 mesh. Used motor oil (with water content, %; 0.13; Density: 872.5 kg/m<sup>3</sup>; Viscosity, mm<sup>2</sup>/s at 40°C: 58, 94; Flash temperature, °C, no less than; 201) obtained from auto maintenance and repair operations and seawater (3.5% salinity) were used in the experiments.

#### 2.2. Method

The sorption system was preceded at room temperature by adding a certain amount of used oil in 1 L seawater (3.5% salinity) within a 2 L glass pot. A total of 0.1 g of biomass was added to the sorption system for time variety (1–10 min) and then separated, drained for 5 min, and re-weighted. Afterward, the wet (oil and water) biomass was dried at 80°C for 24 h. Water sorptions content ( $W_c$ ) (g) and oil sorption content  $(O_c)$  (g) were determined by the subsequent equations:

Water sorption content
$$(W_c)(g) =$$
 Wet biomass $(M_{(W+O)})$   
- Dried wet biomass $(M_{D(O)})$  (1)

Oil sorption content 
$$(O_c)(g) = \text{Dried wet biomass}(M_{D(O)})$$
  
- Dried initial biomass  $(M_F)$  (2)

The oil sorption percent  $(S_p \%)$  was determined by the following equations:

$$S_{p}\% = \frac{\begin{pmatrix} \text{Dried wet biomass} \left(M_{D(O)}\right) - \\ \text{Dried initial biomass} \left(M_{F}\right) \end{pmatrix}}{\text{Dried initial biomass} \left(M_{F}\right)} \times 100$$
(3)

Oil sorption capacity  $q_e$  (g of oil/g of biomass) can be determined from the subsequent equation:

$$q_e\left(\frac{g}{g}\right) = \frac{\text{Oil sorption content}\left(O_c\right)}{\text{Dried initial biomass}\left(M_F\right)}$$
(4)

#### 3. Results and discussion

#### 3.1. Characterization

Agricultural components namely cellulose, hemicelluloses, and lignin are the major constituents of *S. incanum* leaves [22]. These constituents can sorb hydrophilic and hydrophobic liquids owing to the fact that they contain sites of both hydrophilic and hydrophobic and to the porous structure of the fibers. Cellulose components have a great affinity for hydrophilic than hydrophobic substances.

#### 3.1.1. Scanning electron microscope (SEM) analysis

SEM analysis of biomass exposes surface consistency and porosity. SEM analysis of *S. incanum* leaves indicated that the fibrous formation of leaves is observed, with some fractures and pores, which confirmed the existence of the macroporous constitution and increased the probability of the oil sorption by the biomass (Fig. 1a).

#### 3.1.2. Fourier-transform infrared spectroscopy analysis

Generally, oil sorption incorporates the physical and chemical sorption owing to the forces between the biomass surface and the oil/seawater system. In order to investigate the hydrophobic nature of biomass, the functional groups were tested by Fourier-transform infrared spectroscopy (FTIR) analysis. Fig. 1b shows the FTIR analysis (Thermo Fisher Scientific, USA) of acetyl and carbonyl groups (C=O stretching) in the xylene constituent of hemicelluloses structure *Solanum* leaves. The acetyl and carbonyl groups (C=O stretching) in the xylene constituent of hemicellulose structures are assigned at the band of 1,731 cm<sup>-1</sup> [23]. The band at 1,265 cm<sup>-1</sup> refers to the

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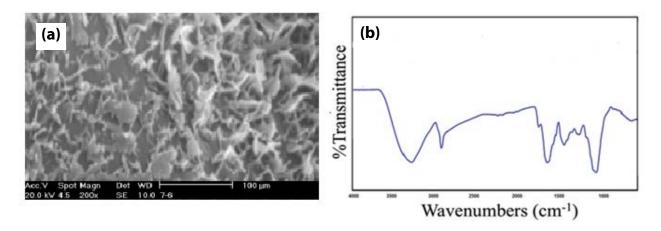


Fig. 1. SEM (a) and (b) FTIR analysis of Solanum leaves.

C–O stretching of the phenolic OH group. The bands at 3,452 and 2,925 cm<sup>-1</sup> are attributed to the OH and C–H groups, respectively. Also, the band range between 835 and 1,505 cm<sup>-1</sup> is attributed to the cellulose structure. The peak at 1,175–1,080 cm<sup>-1</sup> is attributed to the C–O–C ester group [22] which is a type of hydrophobic grouping and is responsible for the hydrophobic nature of biomass.

#### 3.1.3. Wettability analysis

Wettability is an imperative parameter for oil sorption. Water contact angle (W-CA) is commonly distinguished by the wettability of sorbent materials [23]. If the W-CA is more than 90° (114.464°), as revealed in Fig. 2a, thus, the sorbent is hydrophobic due to a large amount of hydrophobic structure present on the biomass surface. Also, the oil contact angle (O-CA) was 51° (Fig. 2b), which indicated that biomass surfaces have excellent ability to sorb oil substance (hydrophilic nature). Fig. 2 shows images of the seawater (Fig. 2c) and oil (Fig. 2d) contact with *S. incanum* leaves, which confirmed the previous results.

#### 3.2. Oil sorption studies

#### 3.2.1. Sorption time evaluation

The efficiency of oil removal was determined with sorption time intervals (1-10 min) at operating parameters 2.5 g oil/1 L seawater and 0.1 g biomass at room temperature (28°C). Fig. 3a shows that there are three sorption sectors in the curve. The first sector is the primary step of oil sorption, which takes place rapidly over the 2 min that is attributed to the significant number of interior pores in the biomass structure. In the next sector, the sorption rate increases slowly from 2 min to an equilibrium state at 4 min. The diffusion rate then turns out to be more difficult due to the decrease in the pore volume in the biomass [23,15]. The final sector represents the stable state where there is no noticeable increase in the oil sorption rate with increasing time. Throughout this period, the biomass tends to be saturated with oil, and extra time will not provide any significant difference in the amount of oil removal. The results reveal higher efficiency of oil removal by Solanum leaves within 4 min.

3.2.2. Biomass dose evaluation

For the determination of oil retention capacity, the amount of oil retained in the *Solanum* leaves after 4 min was utilized as the starting point for calculating oil sorption capacity. The oil sorption capacity of biomass could be influenced by the biomass dose. The biomass doses' effect on the oil removal percent was studied for different doses ranging from 0.1 to 0.6 g in 4.3 g oil/1 L seawater at 4 min and 28°C. Fig. 3b shows that the oil removal percent is increased from 45% to 95% with rising biomass dose from 0.1 to 0.4 g/L, which is due to the increase of sorption sites and the surface area growth in the sorption system [18]. On the other hand, the oil sorption capacity decreased due to the elevated unloaded sorption sites (dose mass), surface area, and remaining unfilled pores throughout the sorption system [24].

#### 3.2.3. Temperature effect evaluation

Temperature is an important factor in the oil sorption process since it affects the viscosity of spilled oil on the surface of seawater. Increasing the temperature of the sorption system leads to a decrease in the oil viscosity and thus increases the oil sorption rate due to easy pores penetration and be imprisoned between surface coarseness until equilibrium state [11,12]; nevertheless, it decreases the sorption capacity as it reduces the time of oil retention on the adsorbent mass during its draining period to separate entrained oil. Fig. 3c shows the effect of temperature on oil uptake. The oil retention capacity reduces from 11.56 to 6.21 g/g with increasing temperature from 28°C to 50°C due to the decrease in the oil viscosity at high temperatures, and the biomass has lost the oil that was trapped in it throughout its draining period [23].

#### 3.2.4. Oileophilicity and hydrophobicity test of biomass

Fig. 3d shows the hydrophobic–oileophilic test of *Solanum* leaves in the oil and seawater systems. According to the results, the oil sorption capacity enhanced with increasing the oil mass in the seawater system, awaiting oil saturation of the biomass was achieved. Furthermore,

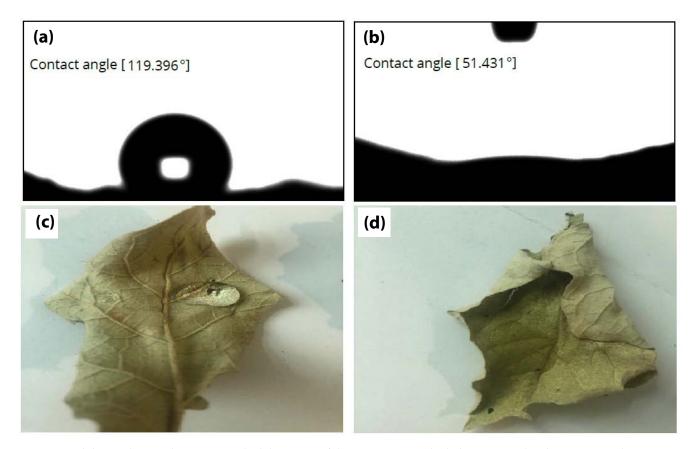


Fig. 2. Wettability analysis with water (a) and oil (b) images of the seawater (c) and oil (d) contact with Solanum incanum leaves.

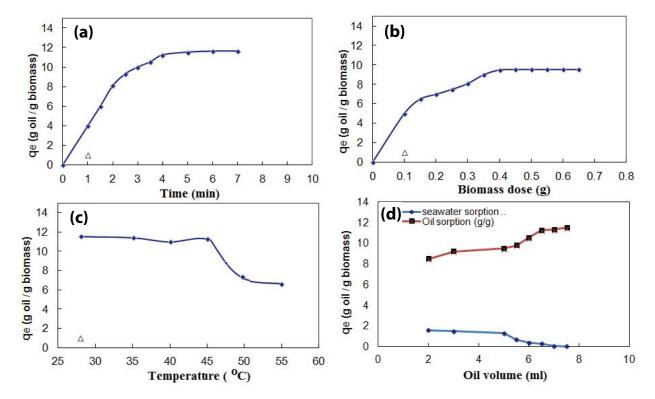


Fig. 3. Sorption dynamics: time effect (a), dose effect (b), temperature effect (c), and oil/seawater sorption capacity (d) of *Solanum* leaves.

the biomass showed small sorption of seawater, which indicates its exceptional hydrophobicity–oileophilicity characteristics. The higher oil selectivity than water indicates that *Solanum* leaves are an excellent biosorbent in the removal of oil spills from seawater.

#### 3.3. Sorption isotherm evaluation

To explain the equilibrium sorption of oil onto adsorbent biomass, Langmuir, Freundlich, and Redlich–Peterson models were investigated by nonlinear systems.

#### 3.3.1. Langmuir isotherm

Langmuir isotherm assumes monolayer homogenous exposure of oil onto sorbent material and is characterized by the non-linear equation [22].

$$q_e = \frac{\left(Q_L K_L C_e\right)}{\left(1 + K_L C_e\right)} \tag{5}$$

where  $C_e$  is the equilibrium oil concentration in (g/L).  $K_L$  (L/g) and  $Q_L$  (g/g) are the Langmuir constants.

#### 3.3.2. Freundlich isotherm

Freundlich model presumes a multilayer heterogeneous sorption of oil onto sorbent and is described by the nonlinear equation [25].

$$q_e = K_F C_e^{\frac{1}{n}} \tag{6}$$

where  $K_F$  (g<sup>(1-1/n)</sup> L<sup>1/n</sup>g<sup>-1</sup>) is the constant of the Freundlich model and *n* is a parameter that refers to the strength of oil sorption onto biomass.

#### 3.3.3. Redlich-Peterson model

Redlich–Peterson isotherm elucidates the homogenous or heterogeneous sorption by the subsequent non-linear equation [26].

$$q_e = \frac{K_{\rm RP}C_e}{\left(1 + PC_e^{\beta}\right)} \tag{7}$$

where  $K_{\text{RP}}$  (L/g) and *P* (L/g)<sup> $\beta$ </sup> are the Redlich–Peterson constants. The exponent  $\beta$  refers to the energy of adsorption.

The non-linear results of sorption isotherms at 30°C are shown in Fig. 4a and Table 1. The results showed that the Redlich-Peterson isotherm provided the greatest fit for the oil sorption process with higher  $R^2$  and lower  $\chi^2$ than other isotherms. Also, the  $\beta$ -value (1.15092) is nearer to one than zero, which denotes that the model is nearer to the Langmuir isotherm than to the Freundlich isotherm as confirmed by the nearer values of correlation coefficient ( $R^2$ ) and chi-square ( $\chi^2$ ) of the Redlich–Peterson isotherm ( $R^2$ : 0.99714 and  $\chi^2$ : 0.03422) and Langmuir isotherm ( $R^2$ : 0.99496 and  $\chi^2$ : 0.06034) than the Freundlich isotherm ( $R^2$ : 0.95097 and  $\chi^2$ : 0.34725) [27]. Therefore, the best fitting of Redlich-Peterson and Langmuir isotherm corroborates the homogeneous monolayer oil sorption process onto sorption sites of biomass rather than heterogeneous multilayer oil sorption [28].

#### 3.4. Sorption kinetic evaluation

The oil sorption kinetics onto biomass is studied by non-linear pseudo-first-order [29], pseudo-second-order [30], and intraparticle diffusion [31] kinetic models. The non-linear form of kinetic models and the results of kinetic parameters are listed in Table 2. The fitting of sorption

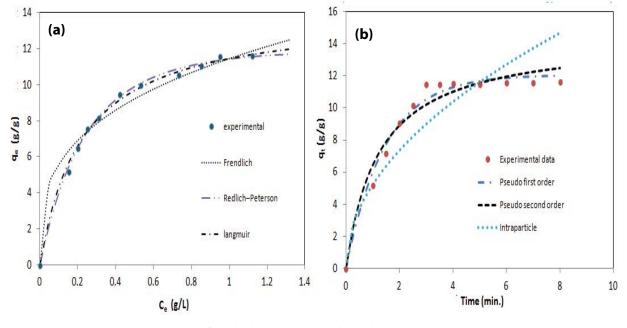


Fig. 4. Isotherm (a) and kinetic (b) models of used oil sorption onto Solanum leaves.

kinetic models with experimental sorption results was appraised by the match between calculated and experimental  $q_{e'}$ ,  $\chi^2$ , and  $R^2$ . According to the non-linear results, the pseudo-first-order kinetic model ( $R^2$ : 0.97941 and  $\chi^2$ : 0.27082) provided good fitting for oil sorption process than the pseudo-second-order ( $R^2$ : 0.95173 and  $\chi^2$ : 0.63476) and Elovich model ( $R^2$ : 0.76666 and  $\chi^2$ : 3.06843) which predict rapid oil sorption and physical nature of oil sorption onto biomass (Fig. 4b).

#### 3.5. Thermodynamics oil sorption

In order to determine the thermodynamic nature of oil sorption onto biomass, the change in entropy ( $\Delta S$ ), free energy ( $\Delta G$ ), and enthalpy ( $\Delta H$ ) were deliberated from the succeeding equations [32,33].

$$\Delta G = \Delta H - T \Delta S \tag{8}$$

Table 1

Nonlinear isotherm parameters of used oil sorption onto *Solanum* leaves

Isotherm model	Parameter	Value
Langmuir isotherm	$Q_{L}$ (g oil/g biomass)	14.00614
	$K_{L}(L/g)$	4.48865
	$R^2$	0.99496
	$\chi^2$	0.06034
Freundlich isotherm	$K_{F} \left( g^{(1-1/n)} L^{1/n} g^{-1} \right)$	11.46553
	п	3.18874
	$R^2$	0.95097
	$\chi^2$	0.34725
Redlich-Peterson isotherm	$K_{\rm RP}$ (L/g)	49.48818
	$P(L/g)^{\beta}$	3.32999
	β	1.15092
	$R^2$	0.99714
	$\chi^2$	0.03422

 $\Delta G = -RT \log K_c \tag{9}$ 

$$K_c = \frac{q_e}{C_e} \tag{10}$$

$$\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$$
(11)

where  $q_e$  is the oil sorption equilibrium capacity (g/g);  $C_e$  is the oil concentration at equilibrium (g/L); *T* is the temperature (°C); *R* is the gas constant (8.314 J/mol K).

Van't Hoff plot (Fig. 5) and the results in Table 3 indicate that the negative value of  $\Delta G$  decreases with the temperature increase from 30°C to 50°C. This further shows that oil absorption is favored at low temperature, which in turn hinders a decrease in the oil's viscosity with a rise in the sorption temperature and hence diminishes the loss of oil absorbed in the biomass during the drainage period. Also, the negative  $\Delta H$  denotes to exothermic of the oil sorption process. The negative  $\Delta S$  value represents the spontaneous sorption of oil onto the biomass [34,35].

#### 3.6. Economic reusability of Solanum leaves

The oil sorption/desorption cycles were conducted to find out the economic reusability of Solanum leaves in the oil spill cleanup. Through squeezing and centrifugation at 500 rpm for 5 min, oil was obtained from loaded biomass. According to Fig. 6, there is a decrease in oil removal percent with sorption/desorption cycles, and Solanum leaves can be reutilized for three oil sorption/desorption cycles before a decrease of less than 50% in the sorption efficiency. The leaves' reusability and oil sorption capacity are evaluated with different agricultural biomasses in Table 4. The leaves' oil sorption capacity (11.56 g/g) was acceptable and noticeably higher uptake than several agricultural biomasses like Sawdust [16], bagasse [15], wheat straw [21], banana peel [2], and barley straw [17]. Nonetheless, other natural sorbents, such as raw kapok fiber [19] and cotton fibers [24], presented higher oil sorption capacity. Biodegradable, cheap,

Table 2 Sorption kinetics models and results for oil sorption onto *Solanum* leaves

Model	Equation form	Parameters	Values
Pseudo-first-order	$q_t = q_e \left( 1 - e^{-k_1 t} \right)$	$K_1$ (L/min): Pseudo-first-order constant	$q_e = 12.07587$ $K_1 = 0.68756$ $R^2 = 0.97941$ $\chi^2 = 0.27082$
Pseudo-second-order	$q_t = \frac{K_2 q_e^2 t}{\left(1 + K_2 q_e t\right)}$	$K_2(g/g min)$ : Pseudo-second-order constant	$q_e = 14.40838$ $K_2 = 0.05626$ $R^2 = 0.95173$ $\chi^2 = 0.63476$
Intraparticle diffusion	$q_t = K_p t^{0.5} + C$	$K_{p}$ : Intraparticle diffusion rate constant C: Constant related to the bounding layer thickness	$K_p = 5.18731$ $R^2 = 0.76666$ $\chi^2 = 3.06843$
$q_e$ and $q_t$ are the oil sorption	on capacity $(g/g)$ at equilibrium and time $t$		

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and available *Solanum* leaves as agricultural solid wastes lead to cost-effective biomass for oil-sorption processes and environmental protection. Moreover, the oil-loaded biomass can be used as a source of energy in many industries.

#### 4. Conclusion

*Solanum* leaves can be used as a biodegradable adsorbent for oil spill cleanup from seawater. The oil sorption capacity reached 11.56 g/g at 4 min, 0.45 g at room temperature. Isotherms and kinetics sorption studies indicated that Redlich–Peterson isotherm and pseudo-first-order models provided the best fitting with the experimental results. Also, the thermodynamic study showed that the oil uptake was an exothermic sorption system and that the use of *Solanum* 

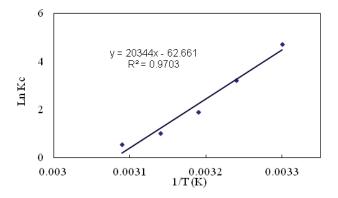


Fig. 5. Van't Hoff plot of used oil sorption onto Solanum leaves.

1.02

0.55

Thermodynamic parameters for used oil sorption onto Solanum leaves

# leaves is favored at lower temperatures to provide the best-desired results. Oil sorption/desorption cycles demonstrated the possibility of economical reuse of *Solanum* leaves in oil spill cleanup up to three cycles before their efficiency decreased by less than 50%. When the results are compared to other agricultural materials, *Solanum* leaves proved to have a competitive efficiency in removing oil from seawater besides their low cost, availability, and biodegradability.

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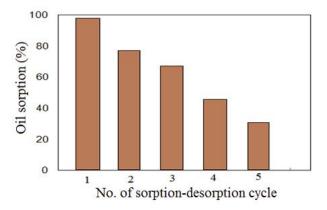


Fig. 6. Sorption-desorption cycles of used oil sorption onto *Solanum* leaves.

 $\Delta S$  (kJ mol<sup>-1</sup> k<sup>-1</sup>)

-0.52

memodynamic parameters for used on sorption onto <i>sounum</i> leaves				
T (K)	$\ln q_e/C_e$	$\Delta G$ (kJ mol <sup>-1</sup> )	$\Delta H$ (kJ mol <sup>-1</sup> )	
301	4.72	-12.65		
308	3.21	-8.98		
313	1.89	-6.38	-169.14	

-3.87

-1.18

318 323

Table 4

Table 3

Comparative of reusability and oil sorption capacity of Solanum leaves with different agricultural sorbents for oil sorption

Sorbent material	Sorption capacity (g/g)	Reusability	Reference
Banana peel	6.35	10	[2]
Bagasse	11.3	5	[15]
Saw dust	6.4	4	[16]
Coir fiber	5.4	6	[16]
Sisal	6.4	6	[16]
Barley straw	7–9.5	_	[17]
Raw kapok fiber	38.1	5	[19]
Wheat straw	4.0	5	[21]
Raw flax fiber	13.75	6	[23]
Raw cotton fiber	30.5	5	[24]
Solanum leaves	11.56	5	Present stud

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