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Article

# A Novel Preparation and Application of Orange Peels Aerogel for Removal of Oil Contaminants in Soils

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## Abstract

Existing soil remediation approaches are either lacking in cost effectiveness, environmental impacts or societal acceptance. Environmental remediation techniques are often characterized by considerable time requirements, and may leave residual effects on the natural ecosystems, thereby potentially compromising net environmental benefits. This study investigated the oil adsorption capacity of aerogels produced from waste orange peels. Aerogels are highly porous three-dimensional materials made from organic and inorganic materials, with low density, and high adjustable specific surface area. Orange peels aerogel was produced from waste orange peels using combined methods of physical, chemical, and thermal modification process, and was dried using freeze-drying method. Adsorption and reusability test were conducted after characterization of the aerogel. Surface characterization of the orange peels aerogel indicated it has an ultra-light density of 0.010417g/cm<sup>3</sup>, high porosity of 99%, and contact angle measurement of 102°. Adsorption experiment was conducted with sandy and clay soils, and the maximum oil adsorption capacities of the orange peels aerogel was 13.55mg/g and 9.60mg/g for sandy and clay soil respectively. High oil adsorption capacity was shown by the produced aerogel and attributed to the ultra-light density of 0.010417g/cm<sup>3</sup> and high porosity of 99% of the orange peel aerogel. In conclusion, the higher oil adsorption capacity of orange peels aerogel in sandy soil than clay soil indicated that soil texture and aerogel properties influenced the oil remediation capacity of orange peel aerogels. The reusability test in three adsorption trials indicated that orange peels aerogel is a sustainable material for the remediation of oil-contaminated soil.

**Keywords:** aerogels; orange peel; oil adsorption; hydrophilic; hydrophobic

## 1. Introduction

Global population is projected to hit 9.8 billion by 2050. As the population grows, the demand for land resources to develop infrastructure, such as housing, and to accommodate human activities increases. This expansion leads to higher extraction of natural resources, greater waste production, and intensified pressure on limited land resources. With increased mining to meet growing needs (Michaux 2021), weak legislation and lack of compliance (Sam et al., 2023), land resources continue to be contaminated. However, existing remediation approaches are time consuming and seldom inefficiently in restoring contaminated sites to meet societal needs and demands (Boukhessaim et al., 2022; Lee et al., 2024). There is also an increased need to integrate a circular economy principle to addressing land contamination to reduce household waste.

Although different remediation methods exist, there are critical limitations. For example, while chemical methods are effective and could engender rapid remediation of hazardous waste, their impact on the natural environment and resource utilization could affect the achievement of sustainable development goals (Vlet et al., 2017; Smith et al., 2019), and its general societal acceptability. Evidence from research indicates that bioremediation could be the most sustainable approach for removing certain hazardous waste (Lee et al., 2023), especially as they are reported to account for almost zero greenhouse emissions, however, they are time consuming, and often unable to biodegrade recalcitrant contaminants (Kästner et al., 2016). Thus, there is an increasing need for rapid, efficient and sustainable remediation approaches (Azuazu et al., 2023).

Aerogels are highly porous three-dimensional materials made from organic and inorganic substances with low density and high specific surface area that can easily be adjusted (Zhu et al., 2017). They are a fascinating type of adsorbents having superhydrophobic and super oleophilic qualities and have demonstrated high sorption capacities (Keshavarz et al., 2021). Studies have shown that aerogels prepared from organic and inorganic materials have been applied in different areas, such as adsorption of contaminants including dyes, heavy metals, and oil from water and air (Zhu et al., 2017; Keshavarz et al., 2021). Aerogels produced from organic sources such as waste biomass are abundant and renewable, reduces dependence on fossil-based chemicals for aerogels production, promoting circular economy practices, lowering greenhouse gas emissions, and encourages the advancement of solvent-free or low-toxicity aerogels systems (Sheesley, et al., 2003; Kelly et al., 2018). The preparation method, structural properties, and performance of cellulose aerogels will largely depend on the biomass type and the amount of cellulose in the biomass. Aerogels prepared from agricultural wastes has received wide application in environmental remediation, due to the amount of cellulose in agricultural wastes (Li et al., 2020; Wang et al., 2020). The fabrication of aerogels from agricultural wastes is inspired by circular economic principles, and the need to reduce amount of waste to landfill (Franco et al., 2021). Also, because most agricultural wastes are cellulose based and abundant in nature, they are cheap materials for fabricating aerogels. Considering that they are soluble in water and organic solvents, this makes them simple and easy to undergo chemical modifications (Tu et al., 2022; Cai et al., 2017; Singh et al., 2017).

Cellulose is high in hydroxyl groups. This justifies the reason most agricultural wastes aerogels do not require crosslinking agent during the preparation phase (Tran et al., 2020; Dai et al., 2023). They are soluble in water and organic solvents making them easier to modify (Long et al., 2018). The drying process is the most critical stage in aerogel preparation. There are two different methods of drying cellulose-based gels, the supercritical fluid method, and the freeze-drying method. The supercritical method usually shows a cauliflower-like structure, while the freeze-drying method shows sheet-like cellulose structure with large, interconnected pores (Franco et al., 2021). Freeze-drying is simple, economical, and an environmentally low risk drying process compared to the other methods and can be used to create a highly spongy aerogel with minimal carbon footprints (Do et al., 2020; Dilamian & Noroozi, 2021; Jumbo et al., 2022). During freeze drying the liquid within the sample is solidified and then transferred under very low pressure by a vacuum pump, making the material spongier than other aerogels with different drying methods (Dai et al., 2022; Dilamian & Noroozi, 2021). The freezing intensity plays key role in the freeze-drying method and can change the consistency of pores (Verma et al., 2020).

According to reports cellulose-based aerogels prepared by the freeze-drying method are among the most promising and studied aerogels for adsorption of contaminants (Nguyen et al., 2019; Wang et al., 2020; Pung et al., 2022). Zhu et al. (2017) stated that aerogels derived from freeze-drying and carbonization processes showed good interconnected mesoporous structure, large specific area of 734.96 m<sup>2</sup>/g needed for adsorption. Also, good structural morphology and adsorption capacities was derived from pineapple fibre and banana stem (Jeng et al., 2020; Tu et al., 2023). The summary of existing studies on agricultural wastes aerogels in Table 1 shows that significant number of agricultural wastes have been successfully modified as cellulose aerogels. However, the results obtained from using waste biomass based cellulose aerogels particularly in contaminants

remediation is variable due to factors such as hydrophilicity and need chemical modification, selective absorption of oil, and poor structural integrity especially in harsh or industrially contaminated environments (Muhammad et al., 2024; Ouyang et al., 2023; Table 1).

There is scarce literature evidence on studies of aerogels that can sustainably remediate contaminated soils due to these limitations. However, the elemental composition of orange peel indicates a promising feature in aerogel for sustainable contaminants remediation in soils (Table 2). As indicated in Table 2, adsorption capacities of orange peels can be significantly improved by modifying their structural properties (Michael-Igolima et al. 2023). In this context, superhydrophobic and oleophilic aerogels derived from orange peels using the freeze-drying method were innovatively applied in a modified adsorption technique to investigate its remediation potency in oil contaminated soils.

**Table 1.** Summary of existing studies on aerogels derived from agricultural wastes.

Waste material	Preparation process	Density g/cm <sup>3</sup>	Porosity %	Contact angle	Sorption capacities g/g	Application	Reference
Banana leaf	Freeze drying	0.14- 0.28	-	-	-	Thermal insulation	Raji et al., 2023
Banana leaf	Freeze drying	0.015	-	149°	35 - 115	Adsorption of oil/organic solvents	Yue et al., 2018
Banana stem	Freeze drying + Pyrolysis	-	-	135°	35	Thermal insulation	Tu et al., 2023
Pineapple leaf	Freeze drying	0.127 – 0.326	97 – 99	-	-	Thermal insulation	Luu et al., 2020
Pineapple leaf	Freeze drying	0.063 – 0.093	92 – 94	-	-	Thermal insulation	Do et al., 2020
Pineapple leaf	Freeze drying	0.013 – 0.033	96 – 99	140°	38	Adsorption of oil	Do et al., 2020
Pineapple leaf + Cotton waste	Freeze drying	0.019 – 0.046	96	-	-	Thermal insulation	Do et al., 2022
Peanut shell	Freeze drying + Carbonization	-	98	141°	27-50	Adsorption of oil/organic solvents	Dai et al., 2022
Seaweed solid waste	Freeze drying + Carbonization	-	-	153°	11-30	Adsorption of oil	Dai et al., 2023
Rice straw	Freeze drying	0.012	99.5	120°	28 - 70	Adsorption of oil/organic solvents	Chhajed et al., 2022
Rice straw	Freeze drying	0.002 – 0.024	98.4 – 99.8	151°	98 - 170	Adsorption of oil/organic solvents.	Dilamian et al., 2021
Rice straw	Freeze drying	0.05 – 0.06	97	150°	13	Thermal insulator	Tran et al., 2020

Bamboo powder + Wastepaper	Freeze drying	0.011	-	118° – 142°	67 – 121	Adsorption of oil	Huang et al., 2022
Soybean stalk	Freeze drying	-	95 – 97	-	16 – 31	Adsorption of oil	Wu et al., 2021
Sugarcane bagasse	Freeze drying	0.016 – 0.122	91.9 – 98.9	-	-	Adsorption of oil/organic solvent	Li et al., 2020
Sugarcane bagasse	Freeze drying	0.016 – 0.112	92 - 99	-	25	Adsorption of oil/organic solvent	Thai et al., 2020
Sugarcane bagasse	Freeze drying	0.012 – 0.108	92.9 – 99.2	140°	23	Thermal insulation/oil adsorption	Kumar et al., 2021
Sorghum stem	Freeze drying	0.146 – 0.167	90	-	-	Adsorption of dyes	Septiani et al., 2019
Sorghum stem	Freeze drying	-	-	-	0.076	Adsorption of organic and inorganic solvents	Chergui et al., 2021
Lupin hull	Freeze drying	0.030	98.1	-	-	Food packaging	Ciftci et al., 2017
Lupin hull	Freeze drying	0.009	99.4	-	-	Food packaging	Deniz Ciftci, 2017
White bamboo	Freeze drying	0.085 – 0.144	90 – 95	114° – 132°	-	Adsorption of oil	Nguyen et al., 2019
Durian	Ultrasound	0.003 – 0.012	99	-	-	-	Xing et al., 2022
Durian	Freeze drying	0.5	-	-	-	Supercapacitor	Wang et al., 2020
Jackfruit	Freeze drying	0.275	-	-	-	Supercapacitor	Lee et al., 2020
Watermelon rind	Freeze drying + Pyrolysis	-	91 – 94	127°	53.51 – 70.52	Adsorption/energy storage	Tu et al., 2022
Pomelo	Freeze drying	-	-	128° – 135°	5 - 36	Adsorption of organic pollutants and oil	Zhu et al., 2017
Pomelo	Freeze drying + Carbonization	0.020	98	132°	49.2 – 71.3	Adsorption of organic pollutants and oil	Shi et al., 2019
Pomelo	Freeze drying	0.18 – 0.23	80 – 87	-	1.7 – 3.9	Adsorption of organic pollutants and oil	Pung et al., 2022
Pomelo fruit peels + wastepaper	Freeze drying	0.611	99	139°	-	Adsorption of oil	Chaudhary et al., 2023
Coconut peat	Freeze drying	28.21	98	-	2.1 – 2.5	Adsorption of oil	La et al., 2021

Coconut peat	Freeze drying		98		24.52	Adsorption of oil	Phat et al., 2022
Coconut fibre	Freeze drying + Carbonization	0.034 – 0.063	96 - 98	-	0.63 – 0.65	Adsorption of dye	Nguyen et al., 2022
Cabbage	Freeze drying	-	-	-	-	Adsorption of oil and organic solvent/ supercapacitor	Cai et al., 2017
Grapefruit peels	Freeze drying	0.051	-	141°	-	Adsorption of oil and organic solvent.	Imran et al., 2020

**Table 2.** Elemental composition of orange peel.

Chemical composition	Mass% (Zapata et al., 2009)	Mass% (Santos et al., 2015)	Mass% This study
Carbon	49.59	44.5	48.67
Hydrogen	6.95	6.1	-
Oxygen	39.7	47.3	36.46
Na	-	-	4.44
Nitrogen	0.66	1.5	-
K	-	-	0.95
Ca	-	-	1.08
Sulphur	0.06	0.4	-
Chloride	0.001	-	8.39
Ash	3.05	4.0	-
Water	2.73	-	-

## 2. Materials and Method

### 2.1. Preparation of Superhydrophobic and Oleophilic Orange Peels Adsorbent

#### 2.1.1. Physical Modification Process

Superhydrophobic aerogels were prepared by combined methods of physical and chemical modification processes. Fresh orange peels sourced locally from a smoothie shop in Bristol, England, were washed in fresh tap water to remove impurities and dried in oven at 75° C for 48hours. The dried peels were ground and sieved to powder using a 300µm sieve. The obtained orange peels powder was used in the chemical modification process.

#### 2.1.2. Chemical Modification Process

According to studies, incorporating relevant chemicals into a biomass improves the structural properties of the biomass and increases the adsorption capacities (Feng *et al.* 2009). Treating orange peels with a base such as NaOH(aq) increases the number of carboxylates and binding sites on the peel by converting methyl esters to carboxylate groups (Feng *et al.* 2009; Liang *et al.* 2009). In this study, 150g of dried orange peel powder was soaked in a mixture of 500ml of (1 mol) NaOH(aq) and 500ml of ethanol in a fume cupboard at room temperature. After 24h, the mixture was decanted and washed with distilled water until PH7 was obtained using a PH meter. Then the sample was dried for 48h in an oven at 75°C to obtain the chemical modified orange peels (CMOP).

### 2.1.3. Thermal Modification Process

Research has shown that chemical treatment increases the number of binding sites on the biomass (Feng et al., 2009), therefore heating the biomass at high temperatures will increase the adsorption capacities of a biomass (El-gheriany *et al.* 2020). Research has shown that calcination of orange peels at temperature range of 400° C to 500° C increase adsorption capacities of the peel, however, the rate of adsorption starts to decrease at temperatures above 500° C (El-Gheriany *et al.* 2020). The thermal modified orange peels were obtained by carbonizing the chemical modified orange peels (CMOP) in a furnace at 500° C for 1hr as previously reported by El-Gheriany et al. (2020).

### 2.1.4. Preparation of Aerogels

To prepare aerogels, 5g of modified orange peels were added to 150ml of water in a glass beaker, and 3g of Oxoid CM0003 Nutrient Agar powder was also added to the beaker. The beaker was placed on a hot plate and stirred continuously with a magnetic stirrer, after about 15minutes the mixture was poured into a plastic beaker and left to cool to obtain an orange peels gel. Drying of the orange peel gel being the most critical step in aerogel preparation was carried out using the freeze-drying technique. For the aerogels to form, the samples were frozen at 0°c for 24hours, then freeze dried for 48hours using a freeze-drying machine and an aerogel is formed. A pictorial summary of different stages involved in the preparation of superhydrophobic, and oleophilic orange peels aerogel is shown in Figure 1.



**Figure 1.** Pictorial presentation of various stages involved in preparing orange peels aerogels.

## 2.2. Characterization of Prepared Aerogel

The prepared aerogel was characterized using FEI Quanta 650 field emission scanning electron microscope and Fourier transform infrared spectroscopy (FT-IR) to study the morphology, and chemical properties of the aerogel. In addition, surface area analysis of the sample was carried out using Keyence digital microscope.

### 2.2.1. Scanning Electron Microscope

The structural morphology of fabricated aerogels was studied with FEI Quanta 650 field emission scanning electron microscope. The samples were coated with gold colored foil to increase their conductivity and mounted with conductive double-coated carbon tape adhesive. The obtained

SEM micrographs of prepared aerogel was studied and was compared with images of other cellulose based aerogels found in literatures.

### 2.2.2. Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR spectroscopy of the aerogels was conducted with FT-IR spectrometer in the range of 400 to 4000cm<sup>-1</sup> to determine the chemical composition of prepared aerogel.

### 2.2.3. Surface Area Analysis

The pore diameter, and the average pore size of prepared aerogels was determined using Keyence digital microscope. Bulk density was calculated using the equation.

$$\text{Bulk density } d = \frac{m}{v} \quad \text{Eq1}$$

Where M is the mass of aerogel measured by weighing the aerogel, V is volume of the aerogel calculated by measuring diameter, thickness, and weight of prepared aerogels.

Porosity was determined manually using the equation 2, as shown in Trans et al. (2020).

$$\text{Porosity (\%)} = 1 - \frac{d}{p} \quad \text{Eq2}$$

Where d is bulk density of the sample and p is true density of crystalline cellulose (1.528g/cm<sup>3</sup>)

## 2.3. Hydrophobic Test

### 2.3.1. The Floating Test

The hydrophobic test of orange peels aerogel was conducted by visual examination using floating test. This involves dipping the aerogel in a transparent glass beaker containing water to observe the extent of floatation exhibited in water.

### Water Contact Angle Measurement

The water contact angle of prepared aerogel was measured using a water contact angle meter (goniometer), three measurements were taken for each sorbent at separate locations on the aerogel to obtain the mean water contact angle of the aerogels.

## 2.4. Adsorption Experiments

Adsorption experiment was conducted in both sandy and clay soil to determine oil adsorption capacities of prepared aerogels in the media. Unconsolidated homogenous mixture of sand and clay soils was packed separately into 2000ml plastic beakers and allowed to consolidate for two weeks. Holes were drilled into the consolidated soil samples up to the bottom of the soil container (both for sand and clay samples), wide enough for the aerogels to enter the hole. 200ml of sunflower oil was poured into a glass beaker, as shown in Table 3. The adsorbents were fixed into the drilled holes, before uniformly spilling the oil on the soil and a contact time of 60minutes was allowed following the ASTM F 726-99 standard method for testing oil spill adsorbents (Figure 2). The aerogel was initially weighed to determine the initial weight before it was put in a perforated paper napkin and placed in open holes created in the soil. The napkin is to prevent the attachment of soil to the aerogel, which may alter oil adsorption result of the Orange peels aerogel (Figure 2). A contact time of 60minutes in the soils (sandy, clay) was allowed for the orange peels aerogel to adsorb more oil. The aerogel was removed from soil, weighed, and the new weight of the aerogel was recorded. The process was repeated in triplicates and recorded in Table 3. The absorbed oil was removed from the aerogel using the squeezing method as reported in Ifelebuegu et al. (2016). Ability of the adsorbent to extract oil was evaluated by investigating the rate of removal, and the adsorption capacities of the aerogels (mg/g) and was obtained using the equation in Imran et al. (2020), as shown in equation 3.

$$Q = \frac{WF - WI}{WI} \quad \text{Eq3}$$

where Q is the sorption capacity of adsorbent, Wi (g) is initial weight of the adsorbent, and WF is the weight of the adsorbent after adsorption.

To ensure the accuracy, reliability, and reproducibility of the collected data, all biosorption experiments were performed in triplicates.

**Table 3.** Physical characteristics of some oil at room temperature.

Oil type	Density (g/cm)	Viscosity (pas)
Vegetable oil	0.910	0.061



**Figure 2.** (a) Orange peels aerogel in sand and clay soil, (b) oil-soaked orange peels aerogel in a napkin.

### 3. Results and Discussion

#### 3.1. Morphology and Structure of the Aerogel

Aerogels are characterised by an open, highly porous, low density, and air-filled structure with high degree porosity and surface area (Anovitz & Cole 2015; Franco et al., 2021). They have very high adsorption capacities due to their peculiar properties (Nita et al., 2020; Franco et al., 2021).

##### 3.1.1. Densities and Porosities

The results of characterisation of the orange peels aerogel were shown in Table 4. From Table 4, the surfaces area has a mean pore size of 58nm, the diameter 51nm, and porosity of 99%. In the table, the aerogel had ultra-light density of 0.010417g/cm<sup>3</sup>, which makes it a good material for adsorption of contaminants. These obtained results (Table 4) corroborate with the research of Luu et al. (2020), Phat et al. (2022), and Cai et al. (2017) where the researchers independently used pineapple leaf, coconut peat, and cabbage-based aerogels for adsorption of oil and concluded that organic aerogels are good oil adsorbents. Table 5 showed absorption properties of aerogel produced from various agricultural biomass waste, however, compared with the results from Table 4, the produced orange peel aerogel has improved properties and more suitable for adsorption of oil from soil having shown higher porosity of 99%, and a minimum adsorption capacity of about 5.3 g/g.

**Table 4.** Surface area analysis of prepared aerogels.

Type of aerogel	Average pore size (nm)	Pore diameter (nm)	Bulk density g/cm <sup>3</sup>	Porosity %	Water contact angle	Adsorption capacity (g/g)
orange	58	51	0.010417	99	102.7	5.3 - 8.0

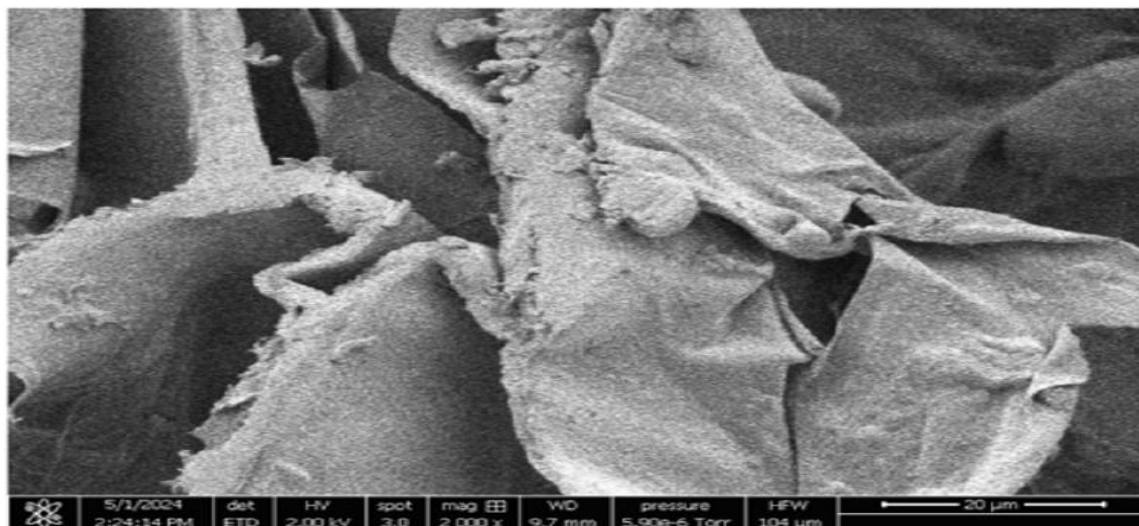
**Table 5.** Summary of the surface area analysis of some cellulose-based aerogels.

Waste material	Preparation process	Density g/cm <sup>3</sup>	Porosity %	Contact angle	Application	Reference
Pineapple leaf	Freeze drying	0.013 – 0.033	96	140°	Adsorption of oil	Do <i>et al.</i> 2020
Peanut shell	Freeze drying + Carbonization	-	98	141°	Adsorption of oil/organic solvents	Dai <i>et al.</i> 2022
Seaweed solid waste	Freeze drying + Carbonization	-	-	153°	Adsorption of oil	Dai <i>et al.</i> 2023
Bamboo powder + Wastepaper	Freeze drying	0.011	-	118° – 142°	Adsorption of oil	Huang <i>et al.</i> 2022
Sugarcane bagasse	Freeze drying	0.016 – 0.122	91.9	-	Adsorption of oil/organic solvent	Li <i>et al.</i> 2021
Sugarcane bagasse	Freeze drying	0.016 – 0.112	92	-	Adsorption of oil/organic solvent	Thai <i>et al.</i> 2020
White bamboo	Freeze drying	0.085 – 0.144	90 – 95	114° – 132°	Adsorption of oil	Nguyen <i>et al.</i> 2019
Pomelo	Freeze drying	-	-	128° – 135°	Adsorption of organic pollutants and oil	Zhu <i>et al.</i> 2017
Pomelo	Freeze drying + Carbonization	0.020	98	132°	Adsorption of organic pollutants and oil	Shi <i>et al.</i> 2019
Pomelo	Freeze drying	0.18 – 0.23	80 – 87	-	Adsorption of organic pollutants and oil	Pung <i>et al.</i> 2022
Pomelo fruit peels + wastepaper	Freeze drying	0.611	99	139°	Adsorption of oil	Chaudhary <i>et al.</i> 2023
Coconut peat	Freeze drying	28.21	98	-	Adsorption of oil	La <i>et al.</i> 2021
Coconut peat	Freeze drying		98		Adsorption of oil	Phat <i>et al.</i> 2022
Grapefruit peels	Freeze drying	0.051	-	141°	Adsorption of oil and organic solvent.	Imran <i>et al.</i> 2020

### 3.1.2. SEM Image of Prepared Orange Peel Aerogel

Scanning electron microscope (SEM) was used to study morphological structure of prepared orange peels aerogels. The image in Figure 3 is a SEM micrograph of orange peels aerogel showing a rough and porous structure. The image in Figure 3 has an average pore size of 51nm (Table 4), which implied a high specific surface area for enhanced adsorption (Shi *et al.* 2019; Pung *et al.* 2022). Figure

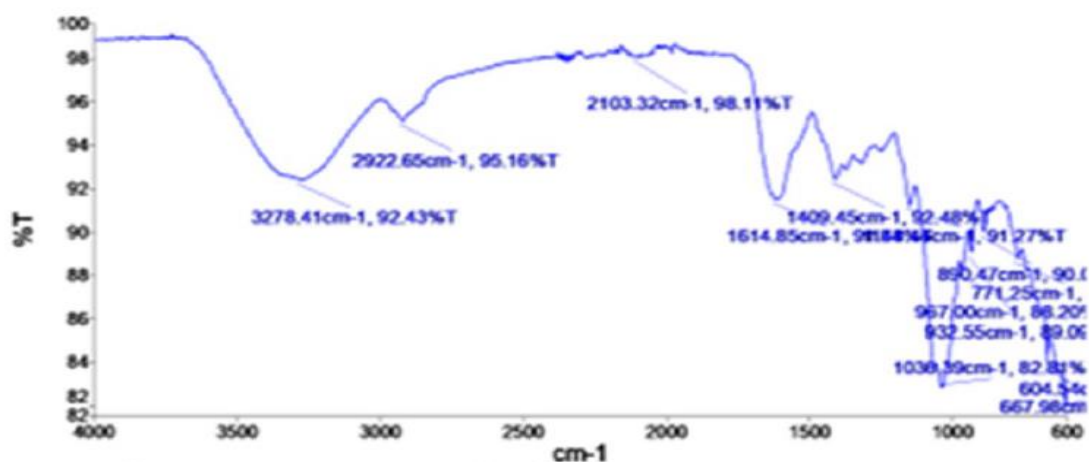
3 has a porous structure that is loosely packed with open spaces which enhanced the adsorption of oil. Similar image has been reported for grapefruits aerogel though with reduced porosity (Imran et al., 2020). High adsorption capacities of aerogels can be attributed to the pore size and density of the aerogel which subsequently enhances adsorption of oil and organic solvents (Huang et al. 2022; Dai et al. 2022).



**Figure 3.** SEM image of orange peels aerogel.

### 3.1.3. FT-IR of Prepared Orange Peel Aerogel

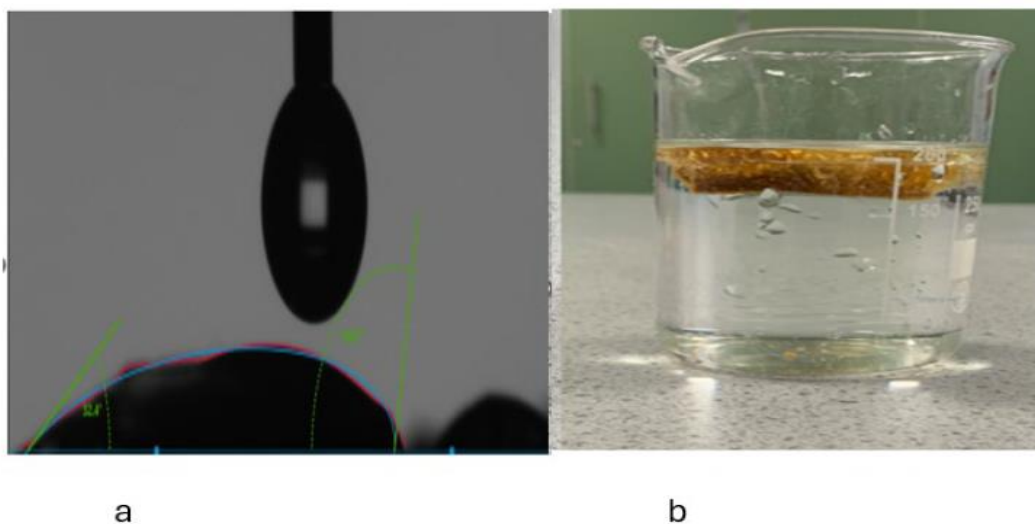
FT-IR spectroscopy was used to analyse chemical composition of prepared orange peel aerogels (Figure 4). The characteristic peak at  $3374.93\text{cm}^{-1}$  which stretches to  $3400\text{cm}^{-1}$  indicated the stretching vibration of hydroxyl groups in orange peels. This was reduced to  $3278.41\text{cm}^{-1}$  in the aerogel sample (Figure 4), which could be linked to the destruction of the hydroxyl functional groups during the freeze-drying process. This finding is corroborated by Do et al. (2020) and Thai et al. (2020) where it was stated that prolonged drying negatively impacts on hydroxyl functional groups during the formation of aerogel. The peak at  $2917.86\text{cm}^{-1}$  shown by the stretching vibration of C-H groups was increased to  $2922.65\text{cm}^{-1}$  (Figure 4). The bands in  $1732.44\text{cm}^{-1}$  and  $1644.52\text{cm}^{-1}$  assigned to the carboxyl and ester groups respectively are present at  $1614.85\text{cm}^{-1}$  and  $1409.85\text{cm}^{-1}$  in the aerogel sample (Figure 4), is attributed to the removal of majority of lignin and hemicellulose after treatment with NaOH. This corroborates reports of Nguyen et al. (2022) and Tu et al. (2022) that pretreatment process by alkalization effectively removes lignin and increases the cellulose.



**Figure 4.** FT-IR image of orange peels aerogel.

### 3.1.4. Hydrophobic Tests of the Aerogel

Water droplets on the sample shown in Figure 5 formed a spherical shape on the surface which was used in measuring water contact angle values of the aerogel (Figure 5). Sessile image of the sample in Figure 5 (a) reveals that water droplets could stand and maintain a spherical shape on the aerogel surface for more than 30secs, showing an average water contact angle of 102.7°C. The water contact angle of a good aerogel should range from 100°C to 160°C, and this range of angle influences the rate of adsorption in cellulose materials (Pan *et al.* 2011; Yue *et al.* 2018). Also, Yue *et al.* (2018) and Do *et al.* (2020) in their research concluded that aerogels with water contact angles higher than 100°C have high adsorption capacities. Thus, the water contact angle value of 102.7°C reported by this study (Figure 5) showed that the orange peels aerogel is a superhydrophobic and oleophilic material. To confirm the hydrophobicity a visual examination using the floating test in water was performed with result shown in Figure 5(b). The aerogel was observed to float in water (Figure 5(b)) with a uniform mirror reflection observed on the surface of the sample which may be caused by formation of interface between entrapped air in the aerogel and the surrounding water due to the surface structure shown in Figure 3 and the presence of the hydroxyl group shown in Figure 4. This corroborates the report of Yue *et al.* (2018) on the surface wettability of banana peels aerogels and wastepaper aerogels where the researchers stated that wettability can be related to presence of the hydroxyl group.



**Figure 5.** (a) sessile image, (b) floating image of orange peels aerogel in a 250ml beaker.

### 3.2. Oil Adsorption of Prepared Aerogels

Results of the oil adsorption test (Table 6) shows that sunflower oil penetrated the orange peels aerogel upon contact with the aerogel and have oil adsorption capacities of 13.55 mg/g in the sand and 9.60mg/g in clay soil. The high oil adsorption capacity of the orange peel aerogel in both soil (sandy and clay) was attributed to the ultra-light density of 0.010417g/cm<sup>3</sup> and high porosity of 99% of the aerogel (Table 4). The investigation further showed that oil adsorption capacity of the orange peels aerogel was more in sandy than in clay soil, which was attributed to the soils texture and properties. This research agreed with the research of Atai et al. (2023) where the researchers stated that remediation of oil vary with soil types.

The oil adsorption capacities of three orange peels adsorbent materials, including pristine orange peels adsorbent, chemical modified orange peels adsorbent, and orange peels aerogel adsorbent in Table 6 showed 60% oil adsorption capacity for the orange peels aerogel, and a 20% oil adsorption capacity for chemical modified orange peels, while the pristine orange peels adsorbent had very low adsorption capacity lower than 1%. The result shows that the orange peels aerogel has the highest oil adsorption capacity and more suited for the remediation of oil in sandy and clay soils. This finding was corroborated by Lv et al. (2023) where the researchers stated that aerogels can accelerate remediation of oil polluted soils.

**Table 6.** Adsorption tests for pristine, chemical modified orange peels, and orange peels aerogel.

Orange peels adsorbents	Oil adsorption capacities (mg/g)			
OPA	1st test	2nd test	3rd test	Average
Dry weight	6.5	6.5	6.5	6.5
Final weight	74	74	73.5	73.83333
Adsorption capacities	10.38	10.38	10.35	10.37
CMOP	1st test	2nd test	3rd test	Average
Dry weight	6.5	6.5	6.5	6.5
Final weight	52	52	51	51.66667
Adsorption capacities	7	7	6.8	6.933333
Pristine	1st test	2nd test	3rd test	Average
Dry weight	6.5	6.5	6.5	6.5
Final weight	6.7	7	6.8	6.833333
Adsorption capacities	0.03	0.08	0.05	0.053333

## 4. Implications of the Study

As transition from fossil fuel dependency to renewable energy sources accelerates globally, a substantial legacy of liabilities persists in many countries, notably numerous oil-contaminated sites left behind by decommissioned and non-decommissioned petroleum operations. These sites are often classified as abandoned and sometimes derelict due to the high costs and resource-intensive nature of conventional remediation methods (Sharma et al., 2018). Developing cost-effective, rapid remediation strategies holds promise for efficiently restoring these sites and facilitating their reuse, ultimately reducing environmental hazards and economic burdens.

This research demonstrates the potential of orange peel-derived aerogels as an innovative and sustainable approach for the remediation of oil-contaminated soils. Leveraging their intrinsic superhydrophobic and oleophilic properties, further enhanced through chemical and physical modifications, these aerogels exhibit high porosity and an extensive array of binding sites, rendering them highly effective for oil adsorption (Deschamps et al., 2003). The deployment of such materials in industrial and environmental remediation practices could introduce an innovative paradigm (Sam et al., 2023), enabling faster and more sustainable land clean-up processes. Moreover, when combined with existing bioremediation techniques, this approach could enhance remediation efficiency,

thereby accelerating the reclamation of contaminated sites in an environmentally responsible manner.

The intrinsic biodegradability of the orange peel aerogel is a critical feature derived from its high cellulose content. As a naturally occurring biopolymer, cellulose exhibits excellent biocompatibility and undergoes biodegradation through microbial activity in the environment. This characteristic ensures that the aerogel materials can be used repeatedly over multiple remediation cycles without accumulating persistent environmental residues. Additionally, at the end of their functional lifespan, these aerogels can be safely degraded in situ, minimizing long-term ecological impacts and aligning with environmental sustainability objectives. Consequently, orange peel aerogels present a sustainable solution not only for effective remediation but also for eco-friendly disposal, reinforcing their role as a circular and environmentally responsible technology.

An additional advantage of this approach is its resourcefulness in reducing orange waste, aligning with circular economy principles aimed at zero waste generation. Transforming orange peels, an abundant agricultural by-product, into functional remediation materials offers a sustainable waste management solution and also significantly reduces volume of waste moved to landfill. Consequently, this strategy could mitigate leachate production from landfills, a major pathway for groundwater contamination, thus contributing to the protection and preservation of critical water resources.

## 5. Conclusions

A superhydrophobic and oleophilic aerogel was successfully produced with orange peel wastes. The produced aerogel had improved surface roughness and porosity alongside increased binding sites by converting methyl esters to carboxyl groups. SEM micrograph of the orange peel aerogel showed a rough and porous structures with high specific surface area of mean pore size of 51nm. The porous structure was loosely packed having open spaces which enhanced oil adsorption of the aerogel thereby increasing its oil remediation capacity. The freeze-drying process reduced the stretching vibration of the hydroxyl groups in the aerogel by 3.6%, therefore in producing aerogel using orange peels waste, there is need for controlled freeze drying to achieve optimum output. Similarly, reduction in the bands of 6.8% and 14.3% for carboxyl and ester groups respectively was found in the aerogel sample which is linked to the removal of majority of lignin and hemicellulose after treatment with NaOH. The sessile image of orange peels aerogel revealed that water droplets could stand and maintain a spherical shape on the aerogel surface for more than 30secs, with an average water contact angle of 102.7°C. A further hydrophobic test conducted by visual examination using the floating test, revealed that the aerogel remained afloat in water indicating that the orange peels aerogel is a superhydrophobic and oleophilic adsorbent. The oil adsorption capacities of the prepared orange peels aerogel were 13.55 mg/g and 9.60 mg/g respectively for sandy and clay soils. The high oil adsorption capacity is attributed to the ultra-light density of 0.010417g/cm<sup>3</sup> and high porosity of 99% of the aerogel. The study concluded that the higher oil adsorption capacity by the orange peels aerogel in sandy soil than clay soil indicated that soil texture and aerogel properties influence the oil remediation capacity of orange peel aerogels. In sum, this study promotes resourcefulness, and reduce reliance on traditional less sustainable remediation methods. The outputs of this study can be adopted in rapid remediation of oil polluted fields and wetlands.

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