

Sorption behaviour of the acetylated *Hibiscus sabdariffa* L. fibre in water and oil

J.M Yelwa^{1*}, *H.A Dihyama*² and *J.T Barminas*²

¹Scientific and Industrial Research Department, National Research Institute for Chemical Technology, Zaria, Nigeria and

²Department of Chemistry, Modibbo Adama University of Technology, Yola, Nigeria

Abstract

The fibre of *Hibiscus sabdariffa* L. was extracted using n-hexane and acetone in the ratio of 1:4 and modified with acetic anhydride in the presence of N-bromo-succinamide as catalyst at a temperature between 35 and 80°C. The study of the sorption behaviour of both the modified and unmodified form of the sample in oil and water was conducted. The sorption studies indicated that the modified fibre has higher oil absorption capacity as compared to the crude form, which indicated that, the hydrophobic property of the modified fibre has increased with the acetylation, thereby enhancing its potential for the use in a non-aqueous absorption processes like oil spill clean-up.

Keywords: Fibre, Sorption, Acetylated, Water, Oil

Full length article *Corresponding Author, e-mail: mjyelwa@gmail.com

1. Introduction

When oil is spilled in water or on land, the physical and chemical properties of the oil will change, progressively. The spilled oil contributes an undesirable taste and odour to drinking water and causes severe environmental damage. Contaminated water cannot be used as a municipal water supply, in industry, or for irrigation [1]. Oil settles on beaches and kills the organisms that lie there. In addition, oil settles on ocean floors and kills benthic (bottom dwelling) organisms, such as crabs. Oil endangers fish hatcheries in coastal areas as well as contaminates the flesh of commercially valuable fish [1].

Effective decontamination and clean-up are necessary after a spill for the protection of the environment and human health. Sorption technique is one of the most effective for the treatment of oil spills. Among the various sorbents that have been employed for oil spill clean-up are synthetic materials such as polypropylene. These are the most commonly used commercial sorbents due to their oleophilic and hydrophobic properties [2]. However, these materials are not biodegradable, which is a major disadvantage. Landfill disposal is environmentally undesirable and incineration is expensive. Therefore, there is a renewed interest in natural sorbents and a wide variety of organic vegetable products such as rice straw, saw dust, cotton, feather, fibres etc. have been employed as sorbents for oil spill clean-up [3].

Using sorbents material to clean-up oil in the marine environment relies on the fact that oil is captured and retained within the structure of the sorbent, reducing the oil's mobility and so minimising the area covered by spills. Binding oil to the sorbent also reduces the oil's initial velocity, thereby reducing contamination of water and sediments by the lighter, more toxic species of hydrocarbon present in oil (e.g polyaromated hydrocarbon etc.) [4].

2. Materials and methods

2.1. Sample preparation and treatment

The *Hibiscus sabdariffa* fibre was collected from Gashala Guw, village in Hong local government Area of Adamawa State. The plant was collected using cutlass as it matures and the stem was separated from other parts of the plant by the use of sharp knife. The fibre was then removed from the stem gently while it was still fresh. Some of the fibre was removed from the stem after the plant has been dried. This was achieved by soaking the plant material in water for three to five days (3-5days), it was then removed and the stem separated from the fibre while it was wet. The fibre was washed thoroughly with water to remove dust, fungus, water soluble components and other foreign materials. The washed fibre was dried in sun light for 12h (4h for three days) and then left to dry at 65°C in an oven. The fibre was reduced in size by the use of piston and motor and then sieved through 20 and 25 British standard sieves (BSS sieves).

2.2. Soxhlet extraction

The plant fibre was extracted by following the method described by Dihyama [5].

2.3. Acetylation procedure

The acetylation of the plant fibre was done by following the method described by Dihyama [5].

2.4 Water absorption test

The raw and modified forms of the fibre were subjected to water absorption test. The samples were immersed in water and allowed to absorb water for specific soaking times, 20, 40, 60 and 80 minutes respectively. The wet sorbents were removed from the water after the specific times and drained on a filter paper for about 10 minutes. The water sorption capacity in gram/gram was calculated using the relation:

$$\text{Water Absorption Capacity (WAC)} = \text{AW} - \frac{\text{AD}}{\text{AD}}$$

Where AW is the amount of wet sorbent and AD is the amount of dry sorbent. The amount of wet and dry sorbent was determined through weighing. The amount of dry sorbent can also be given by amount of wet sorbent (AW) – weight of sorbent before soaking.

That is:

$$\text{AD} = \text{AW} - \text{Weight of sorbent before soaking}$$

2.4.1 Study of the oil sorption behaviour

The oil sorption property of the modified and unmodified form of the *Hibiscus sabdariffa* fibre was tested by placing 500mg each of modified and unmodified form of the *Hibiscus sabdariffa* fibre in a 250 ml beaker containing about 5g of oil displaced in 100 ml of water at room temperature or slightly above room temperature. The sample was left in the mixture for about five (5) minutes with gentle stirring in the first two (2) minutes. The sample was then removed from the mixture using sieve net, and the net was then dried in an oven at about 60°C for about thirty (30) minutes. The amount of oil and water absorbed by the sample was then calculated from the information obtained from the experiment.

2.4.2. Swelling ability (S) and anti-swelling efficiency (ASE) tests

The swelling ability was determined as adopted by Nwankwere *et al.*, [4] using sets of 500g of the plant material each place in a separate 10ml measuring cylinders, then 5cm³ of distilled water was added, there was a replacement of water daily for five days. The sample was weighed and the adsorption (or swelling ability) values were calculated according to the equations below after each water replacement in line with the formula:

$$S(\%) = [(W_{\text{wet}} - W_{\text{dry}})/W_{\text{dry}}] \times 100$$

Where S (%) = swelling ability percentage, W_{wet} = weight of wet plant material, W_{dry} = weight of dry plant material, Mo = mass of bottle (measuring cylinder) and Vo = volume of bottle (measuring cylinder)

3. Results and discussion

3.1. Water sorption behaviour of the crude and modified (acetylated) fibre

As it is expected of most plant materials, the study of the water sorption behaviour of the fibre before and after the treatment indicated that, the crude fibre has high water sorption behaviour and very low oil sorption behaviour. This is due to the characteristic hydroxyl (OH) functionality of the crude fibre which confers water sorption behaviour on them. On the other hand, the modified form of the fibre indicated a very poor water sorption capacity due to the removal of the water attracting centres on them by the acetylation modification process, hence the introduction of the water hating moiety (the acetyl group) which makes them to have a very low affinity for water, hence, has very low water absorption behaviour [4]. As indicated by the mathematical expression below:

$$\text{Water absorption capacity (WAC)} = \text{AW} - \text{AD}/\text{AD}$$

Where AW stands for amount of wet sorbent and AD stands for amount of dry sorbent. The water sorption capacity of both modified and unmodified fibre is presented in the figure 1 below.

3.1.1. Effects of temperature on the water sorption behaviour

Varying the condition of temperature of water, may result in a corresponding change in the rate or extent to which it will be absorbed or adsorbed by a particular sorbent.

3.1.2. Effects of time on the water sorption behaviour

The effect of time on the water sorption property of the sorbent material is that, the higher it stays in the water, the more it will adsorb the water to its surface or absorb it into its body to its maximum capacity or saturation level. For a given sorbent material, there is a specific soaking time it requires for it to start absorbing water or any other thing and also a time when it will reach a saturation level under normal condition.

3.2. Oil sorption behaviour of the crude and the modified (acetylated) fibre

The principal aim of the study of the medication process of the *Hibiscus sabdariffa* fibre is to alter its water sorption property and increase its oil sorption property. And all the result obtained in this work with respect to the sorption behaviour of the modified form of the fibre, indicated significant changes in the sorption behaviour in which the water sorption ability has reduced in case whereas the oil sorption capability has increased. The oil sorption

study of the treated and the crude form of the *Hibiscus sabdariffa* fibre indicated a positive change in respect to the absorption behaviour of the fibre after the treatment. The crude fibre shows a very low oil sorption capability, whereas the modified form of the fibre indicated a significant increase in the oil sorption capability. This is as result of the introduction of the hydrophobic centre (CH₃COO) to the modified fibre which eventually, increases its oil attraction behaviour and consequently, reducing its oil attracting behaviour [6]. The result of the oil sorption behaviours of both modified and unmodified fibre are presented on table 2.

3.2.1. Effects of temperature on the oil sorption behaviour

Change in temperature affects almost every process, being chemical, physical, biological, mechanical and so on. The effect of change in temperature on the sorption of oil by the modified *Hibiscus sabdariffa* fibre is related to the effect on the oil itself as well as the sorbents. As temperature increases, the viscosity of the oil will be reduced and consequently increase the rate of flow of the oil

and the absorption rate will also be increase. For volatile oil, increase in temperature also lead to the increase in the oil's volatility and hence increases the rate at which it will be absorbed by the sorbent.

3.2.2. Effects of time on the oil sorption behaviour

The variation of the oil sorption behaviour of a *Hibiscus sabdariffa* fibre with time was studied by soaking the sorbent in a 250ml beaker containing 5g of oil displaced in 100ml of water, for specific soaking times; 20, 40, 60, 80, 100 min., the result shows increase in the sorption with time.

3.3. Oil/water sorption behaviour of both modified and unmodified form of the fibre

Figure 3 below show the sorption capacity of modified and unmodified fibre at different weight at constant time (20 minutes). It can be seen that the oil/water sorption capacity of the modified fibre has significantly increased as compared with the unmodified fibre. It was also observed that as the weight of the sorbent increases, it sorption capacity also increases.

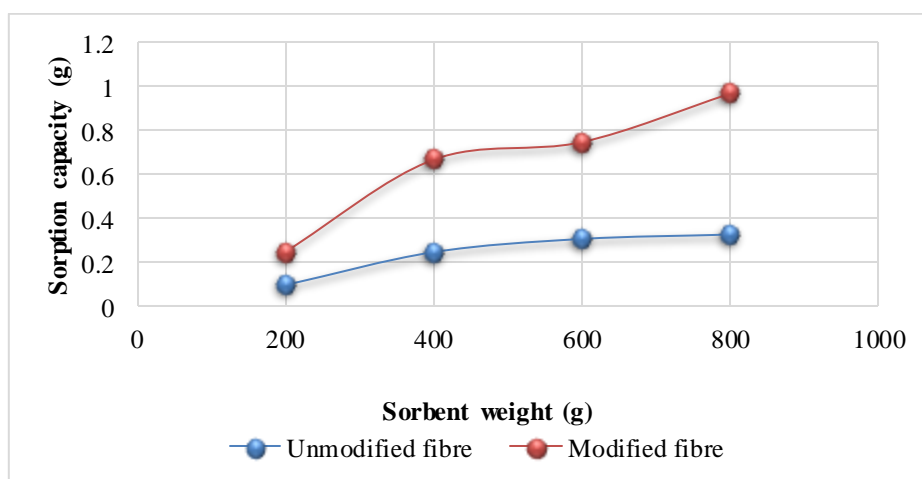


Figure 1. Water sorption capacity of modified and unmodified fibre

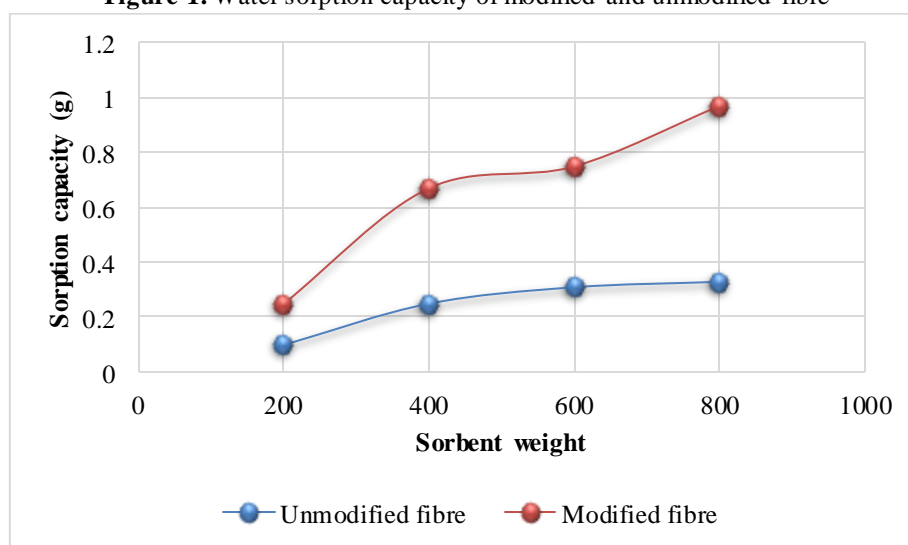


Figure 2. Oil sorption capacity of modified and unmodified fibre

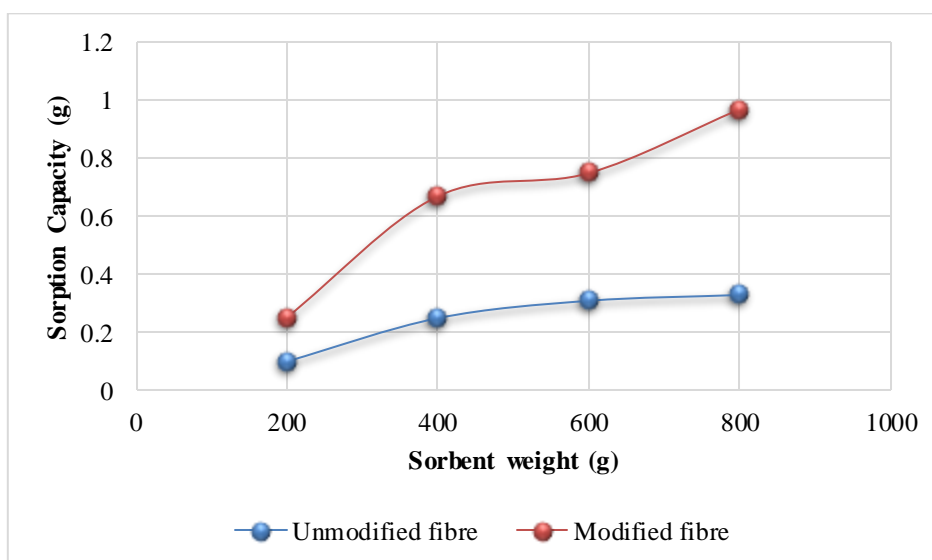


Figure 3. Oil/water sorption behaviour of both modified and unmodified form of the fibre

3.4. Statistical analysis of some properties of the fibre

The observed effects of change in catalyst, time and temperature on degree of acetylation of the *Hibiscus sabdariffa* fibre were tested for statistical difference using the analysis of variance (ANOVA). The result shows that, time effect is non-significant on the extent of acetylation of the *Hibiscus sabdariffa* fibre, but catalyst and temperature had significant effects.

Conclusions

The hydrophobic treatment of a plant such as *Hibiscus sabdariffa* fibre is a suitable method for producing an environmentally friendly, low cost, less hazardous and biodegradable sorbents for oil spill clean-up in aqueous environment, due to its hydrophobic and oleophilic properties. This plant derived sorbent can replace the synthetic sorbents due to its biodegradability, availability, less hazardous nature unlike the synthetic sorbents materials which are expensive and non-biodegradable.

Reference

[1]. M. Hussein, A.A., Amer, and I.I. Sawsan (2008). Oil spill sorption using carbonised pith bagasse trial for

I

practical application. *International Journal of Environmental Science and Technology*. 5(2): 223-242.

- [2]. C. Teas, S. Kalligeros, F. Zanikos. S. Stounas, E Lois, and G Anastopolous (2010). Investigation of the effectiveness of adsorbent materials in oil spill clean-up. *Desalination*. 140(3): 259-264.
- [3]. C. Nwilop. and O.T., Badejo (2010). Impacts and management of oil spill pollution along the Nigeria coastal areas.
- [4]. E.T, Nwankwere, C.E., Gimba, J.A. Kagbu. and B.K. Nale (2010). Sorption studies of crude oil on acetylated rice husks. *Archives of Applied Science Research*. 2: 142-145.
- [5]. H.A, Dihyama, (2017). Thermodynamics of thermodynamic studies of acetylated *Hibiscus sabdariffa L.* fibre for use as oil sorbents. Unpublished masters Thesis. Modibbo Adama University of Technology, Yola, Adamawa.
- [6]. R.F., Johnson and J.E. Halligan (1973). Removal of oil from water surfaces by sorption on unstructured fibres. *Environmental science and Technology*. 7: 339-443.