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Acetylation of banana fibre to improve oil absorbency

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ABSTRACT

Oil spill leaves detrimental effects on the environment, living organisms and economy. In the present work, an attempt is made to provide an efficient, easily deployable method of cleaning up oil spills and recovering of the oil. The work reports the use of banana fibres which were acetylated for oil spill recovery. The product so formed was characterized by FT-IR, TG, SEM and its degree of acetylation was also evaluated. The extent of acetylation was measured by weight percent gain. The oil sorption capacity of the acetylated fibre was higher than that of the commercial synthetic oil sorbents such as polypropylene fibres as well as un-modified fibre. Therefore, these oil sorption-active materials which are also biodegradable can be used to substitute non-biodegradable synthetic materials in oil spill cleanup.

1. Introduction

Oil is one of the most important energy and raw material sources for synthetic polymers and chemicals worldwide (Annunciado, Sydenstricker, & Amico, 2005). During production, transportation, storage and usage of oil there is always a risk of oil spillage. Oil spill occurs over the seas, water bodies and land surfaces due to ship breakage, tanker disasters, wars, operation failures, equipment breaking down, accidents and natural disasters. Oil spills into land, river or ocean impose a major problem on the environment (Azzam & Madkour, 2008; Wei, Mather, Fotheringham, & Yang, 2003). Under favorable conditions, oil in water body continues to spread over the water surface and forms a monomolecular layer and affects the natural habitat. Oil film which is formed on the surface of water due to spills impairs the exchange of energy, heat, moisture, and gases between the water reservoirs and the environment. This leads to considerable harm to environment and marine life. Oil spills harm the beauty of polluted sites; give strong odour and the excessive growth of green algae, alter sea colour and the landscape (Annunciado et al., 2005). Viscosity of the oil also plays an important role in deciding the severity of such spill. Viscous oils spread more slowly than less viscous ones and therefore, water temperature, along with wind speed and sea conditions have tremendous effect on the extent of oil spreading. It is reported that spreading is important in determining the fortune of spilled oil (Reed et al., 1999; Wei et al., 2003) through evaporation, emulsification and neutral dispersion.

One of the ways to clean up the oil spills is to make use of proper sorbents. The sorbents presently in use can be classified as polymers, natural materials or treated cellulosic materials. Most commonly used commercial sorbents are synthetic sorbents made of polypropylene or polyurethane (Deschamps, Caruel, Borredon, Bonnin, & Vignoles, 2003). They have good hydrophobic and oleophilic properties, but their non biodegradability is a major disadvantage (Choi & Cloud, 1992; Deschamps et al., 2003). A number of natural sorbents have been studied for use in oil-spill cleanup, e.g. cotton (Choi, 1996; Johnson, Manjrekar, & Halligan, 1973), wool (Radetic, Jocic, Jovancic, Petrovic, & Thomas, 2003), bark (Haussard et al., 2003) and rice husk (Nwankwere, Omolaoye, Nwadiogbu, & Nale, 2011). These agricultural products and residues are locally available and inexpensive. Some are waste materials and hence their reuse will result in savings in disposal fee. Cellulose is an abundant and naturally occurring polymer that can be obtained from numerous resources. Its structure is organized into fibrils, which are surrounded by a matrix of lignin and hemicellulose (Rosa et al., 2010). The cellulosic products which exist in fibrous form can be easily formed into mats, pads and non-woven sheets for convenient applications (Fanta, Burr, & William, 1986).

One such cellulosic fibre is banana fibre obtained from banana plants. These banana plants are of the family Musacease and are cultivated primarily for their fruit. As such, after harvesting the fruit, the matured pseudostems are generally disposed as a landfill or left to decompose slowly in a plantation field (Bilba, Arsene, & Ouensanga, 2007). Banana fibre can also be extracted from the "pseudostems" which is clustered, cylindrical aggregation of leaf

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stalk bases. Banana fibre is a ligno-cellulosic fibre which has relatively good mechanical properties. The extraction of fibre however is not a common practice.

The objective of this work is to provide an environmentally acceptable method of cleaning up oil spills and also to get an applicable technique which allows its recovery using banana fibre as a substrate. This work evaluates the use of banana fibre for sorption of spilled oil from aqueous media. Since banana fibre tends to also absorb water, its oil sorption capacity may be drastically reduced. Thus, esterification is carried out to increase the hydrophobicity of the fibres and also to enhance oleophilicity.

2. Experimental

2.1. Materials

Banana fibre was purchased from a local market (Mumbai, India) and was dried in sunlight. Acetic anhydride, N-Bromosuccinimide (NBS) and all other chemicals were supplied by S.D. Fine Chemicals Mumbai India. High density oil used for the testing purpose was purchased from local market.

2.2. Methods

2.2.1. Acetylation in a solvent free system

A fixed quantity of banana fibre (10 g) was placed in a 500 ml round bottom flask containing required ml of acetic anhydride and N-Bromosuccinimide (NBS) (2%) as a catalyst. The flask was then placed in an oil bath set at the required temperature (120 °C) using atmospheric pressure with a reflux condenser fitted. After the reaction for the required time (1 h) the flask was removed from the oil bath and the hot reagent was decanted-off. The banana fibre was then thoroughly washed with ethanol and acetone to remove the un-reacted acetic anhydride and acetic acid by-products. The modified fibres were then dried in an oven at 60 °C to constant weight. The oven-dry materials were weighed to determine the weight gains on the basis of initial oven-dry weight (Sun, Sun, & Sun, 2004). To confirm the results, each experiment was repeated thrice under the same conditions and the weight percent gain (WPG) values were estimated which were well within the acceptable standard deviation of 0.2%. Weight percent gain of the banana fibre due to acetylation was calculated using the formula:

$$\text{WPG} = \frac{W_2 - W_1}{W_1} \times 100$$

wherein, W_1 and W_2 are the weights of banana fibre and acetylated banana fibre, respectively.

2.2.2. Oil absorptivity

Oil absorptivity was determined by using method reported in literature (Sun et al., 2004). A fixed quantity of machine oil (50 g) was suspended in water in a beaker. The acetylated banana fibre (1 g) was added at room temperature and allowed to absorb oil for 1 h. The banana fibre was then picked up and held to drain-off the excess amount of oil. The banana fibre was then reweighed to determine the oil absorptivity.

2.2.3. Recovery of sorbed oil and reusability of sorbents

In order to examine the reusability of these sorbents, methods described elsewhere were followed, with the limitation that this method gives only an approximate value of oil sorption. The method is briefly summarized. Machine oil (50 g) was suspended in water in a beaker. The acetylated banana fibre (1 g) was added and mixed for 1 min at room temperature and allowed to absorb oil for 1 h. The sorbent with oil was weighed and then squeezed between two rollers at a pressure of 10kgf/cm before it was reweighed to determine the amount of recovered oil. The squeezed sorbent was again used in the sorption process as before (Choi & Moreau, 1993). The efficiency of sorbent reusability was determined by oil sorption capacity of each sorbent after repeated sorption and mechanical desorption cycles (Ansari, East, & Johnson, 2003).

3. Chemical characterization

3.1. IR spectra

The IR spectra of original and acetylated banana fibre samples were recorded using FTIR spectrophotometer (Shimadzu 8400s, Japan) using ATR sampling technique by recording 45 scans in % T mode in the range of 4000–600 cm^{-1}.

3.2. TGA

Thermal gravimetric analysis (TGA) of the unmodified and acetylated banana fibres was carried out by regular method. The thermograms of samples were recorded on Shimadzu 60H DTG machine using aluminum pan between temperature range 30–550 °C and under the inert atmosphere of N_2 at a flow rate of 50 ml/min.

3.3. Degree of acetylation

The percent acetylation (acetyl %) was determined using titration method. Acetylated banana fibre (1 g) was placed in a 250-ml flask, and to this, 50 ml of 75% ethanol in distilled water was added. The loosely stopper flask was agitated, warmed to 50 °C for 30 min, cooled, and 40 ml of 0.5 M KOH was added to it. The excess of alkali was back titrated with 0.5 M HCl with phenolphthalein as an indicator. The solution was allowed to stand for 2 h and then any additional alkali which may have leached from the sample was titrated (S_R). A blank reading (B_R) using the original unmodified banana sample was taken.

$$\text{Acetyl\%} = \frac{\{(B_R - S_R) \times \text{molarity of the HCl} + 0.043 \times 100\}}{\text{Sample weight}}$$

B_R and S_R are titration volumes in ml and sample weight is in grams on dry weight basis.

Degree of substitution (DS) was calculated as reported in literature (David, Huijun, Duohai, & Harold, 1999).

$$\text{DS} = \frac{(162 \times \text{acetyl\%})}{\{4300 - (42 \times \text{acetyl\%})\}}$$

3.4. Scanning electron microscopy (SEM)

Analysis of the morphology of dried and modified sample was carried out using scanning electron microscope (JEOL, Japan), from Institute of Chemical Technology. The samples were sputter coated with gold layers and images were recorded using scanning electron microscope.

3.5. Chemical analysis

An accurately weighed 2 g of banana fibre sample was boiled in ethanol (4 times) for 15 min, washed thoroughly with distilled water and kept in oven for drying at 80 °C for overnight. It was again weighed and then divided into two equal parts in which one part is considered as A fraction. Second part of residue was treated with 24% KOH for 4 h at 25 °C, washed thoroughly with distilled water and dried at 80 °C over night and the dry weight was taken as B fraction. The same sample was further treated with 72% H_2SO_4 for 3 h to hydrolyse the cellulose and then refluxed with 5% H_2SO_4 for 2 h.

H_2SO_4 was removed completely by washing it with distilled water. It was then dried at 80 °C in oven for overnight and dry weight was taken as C fraction (Moubasher, Abdel-Hafez, & Mohanram, 1982).

Cellulose = B-C.
Hemicellulose = A-B.
Lignin = C itself.

4. Results and discussion

At the outset, experiments to optimize various parameters of acetylation of the banana fibre such as time, temperature, catalyst concentration, solid to liquid ratio etc. were undertaken and the results of this set of experiments are given in Table 1. Since the aim of the modification of the fibre by acetylation is to get an oil absorbent product, optimization parameter were related to those conditions of acetylation, resulting in the product giving maximum oil absorbency. The influence of individual parameters of acetylation reaction which, in fact makes the fibre hydrophobic and oleophilic are discussed further.

4.1. Effect of time

In the beginning, in order to optimize time, the reaction time was varied using other parameters of reaction fixed as shown in Table 1. The results indicate that with increase in time from 0.5 h to 1 h, the WPG of acetylated fibre was found to increase, however further increase to 2 h, it did not reflect in increase in WPG showing leveling-off in accessibility of banana fibre for the reactants indicating near stagnancy in level of acetylation. It is to be noted that banana fibre being lignocellulosic, when subjected for acetylation reaction, depending upon the accessibility of the cellulose which takes part in the reaction, the —OH groups in cellulosic structure get converted into —O CO CH_3 (acetate groups), which are bulkier as well as of higher molecular weight.

$$\underset{\text{Lignocellulose}}{\text{LC-OH}} + \underset{\text{Acetic Anhydride}}{R_2(C{=}O)_2O} \rightarrow \underset{\text{Acetate}}{\text{LC-O(C=O)R}} + \underset{\substack{\text{Byproduct}\\ \text{Acetic Acid}}}{\text{RCOOH}}$$

Hence on acetylation the modified sample is expected to give increase in the weight of the product as reported in WPG and also increase in accessibility. With increase in time initially, extent of acetylation increases. Whereas normal banana fibre shows 2.10 g/g oil absorption, the acetylated samples showed tremendously enhanced extent of oil absorption, which was due to increased hydrophobicity and enhanced oleophilicity of the modified fibre sample. Beyond 1 h reaction time, however, there was no appreciable change in oil sorption, although slight weight gain was observed. Hence 1 h reaction time was taken as an optimum time for acetylation as it gave near maximum level of oil sorption.

4.2. Effect of temperature

Using optimum time of reaction as 1 h and maintaining other parameters fixed as shown in Table 1, the temperature of reaction was varied. The WPG increased significantly with the increment of reaction temperature. This could be attributed to the increased compatibility of the reaction ingredients and swellability of the banana fibres and thus increased accessibility of it to the reactants at higher temperature (Khalil, Hashem, & Hebeish, 1995). Since the hydroxyl groups of the cell wall polymers form extensive hydrogen bonding networks within the matrix and during the acetylation process the reaction of the acetic anhydride with hydroxyl group requires the breaking of these original hydrogen bonds (Satchell, 1963), increase in temperature favors the fibre swelling as the reaction proceeds. In other words increasing temperature favored the breaking of hydrogen bonds, swelling of the fibres and diffusion of the esterifying agent, thus enhancing the extent of modification. However the WPG at 140 °C temperature was only marginally higher than that at 120 °C temperature. The oil absorption level was also slightly increased. Hence considering thermal energy required and economy of the operation, 120 °C temperature was taken as an optimum temperature.

4.3. Effect of catalyst on WPG

Using optimized time and temperature of reaction as fixed parameter, and keeping solid to liquid ratio also fixed till it was optimized; the quantity of NBS was varied as shown in Table 1. The characteristic properties of the modified product in terms of WPG and oil sorption level were estimated and the results are presented in the same table. The increase in quantity of NBS substantially accelerated the extent of acetylation and use of 2% NBS (2 g NBS in 100 ml acetic anhydride) as a catalyst at 120 °C for 1 h resulted in an increase in the WPG by 13.89% which was highest. Consequently the oil absorption of this sample was maximum and hence 2% NBS catalyst is considered to be an optimum one for acetylation process. The role of NBS is not clear but a plausible explanation is that NBS might act as a source for Br^+, which in turn activates the carbonyl groups of acetic anhydride to produce the highly reactive acylating agent (CH_3—CO—N—($OCCH_2CH_2CO$—). This acylating agent reacts with hydroxyl groups of banana, which upon elimination of NBS produces acetylated banana fibre (Banana fibre—O—CO—CH_3) (Sun et al., 2004). This hypothesis, however, needs further investigation.

4.4. Effect of solid to liquid ratio on WPG

Finally using all these optimized parameters as fixed ones, solid material, i.e. fibre weight to liquid volume was varied. The reaction was then carried out and product was analysed for WPG and oil sorption. The results indicate that when solid to liquid ratio was varied from 1:10 to 1:20, the extent of acetylation increased which was mainly because of increased amount of reactant liquor available for reaction with solid fibre enhancing its proper penetration inside the fibre. However, beyond this ratio, at 1:30 ratio there was only marginal increase in the extent of acetylation as seen in WPG as well as in extent of oil absorption. Hence solid to liquid ratio of 1:20 was considered to be an optimum one.

4.5. FT-IR analysis

The FT-IR results shown in Fig. 1 indicate that banana fibre undergoes modification by means of acetylation reaction. The peak at 1750 cm^{-1} is because of the introduction of ester group which is present in the acetylated sample, which otherwise is absent in raw banana fibre. Similarly, the reduction of the intermolecular hydrogen bonding between 3406 and 3471 cm^{-1} confirms the introduction of the acetyl groups in the banana structure thus replacing the hydroxyl groups. It is worth noting that an acetylation of plant fibres reduces the hygroscopic nature of the cell wall and the incorporation of such acetylated fibres into plastics enhances weather resistance and dimensional stability of the composites (Sun and Sun, 2002).

4.6. Thermal analysis

Fig. 2 shows the thermo gram of raw and acetylated banana. In the initial stage weight loss of raw and acetylated samples were 7.39% and 10.59% at 250 °C respectively; beyond this temperature the drastic decomposition of the sample resulted into the significant weight loss which was 35.67% for raw banana fibre and 65.18%

Table 1
Effect of different parameters of acetylation on oil absorption.

Sr. No.	Time (h)	Temperature (°C)	Catalyst (%)	Solid to liquid ratio[a]	WPG (%)[b]	Oil absorption (g of oil/g of fibre)
1. Effect of time						
A	0.5	120	2	1:20	10.25	15.73
B	1	120	2	1:20	13.89	18.12
C	2	120	2	1:20	14.08	18.09
2. Effect of temperature						
A	1	100	2	1:20	9.12	13.64
B	1	120	2	1:20	13.89	18.12
C	1	140	2	1:20	14.14	18.37
3. Effect of catalyst concentration						
A	1	120	1	1:20	9.65	12.15
B	1	120	2	1:20	13.89	18.12
C	1	120	3	1:20	13.84	17.56
4. Effect of solid to liquid ratio						
A	1	120	2	1:10	9.40	11.86
B	1	120	2	1:20	13.89	18.12
C	1	120	2	1:30	13.47	17.92

[a] Solid to liquid ratio represents ratio of dried banana (g) to volume (ml) of acetic anhydride.
[b] WPG represents the weight percent gain of banana due to acetylation.

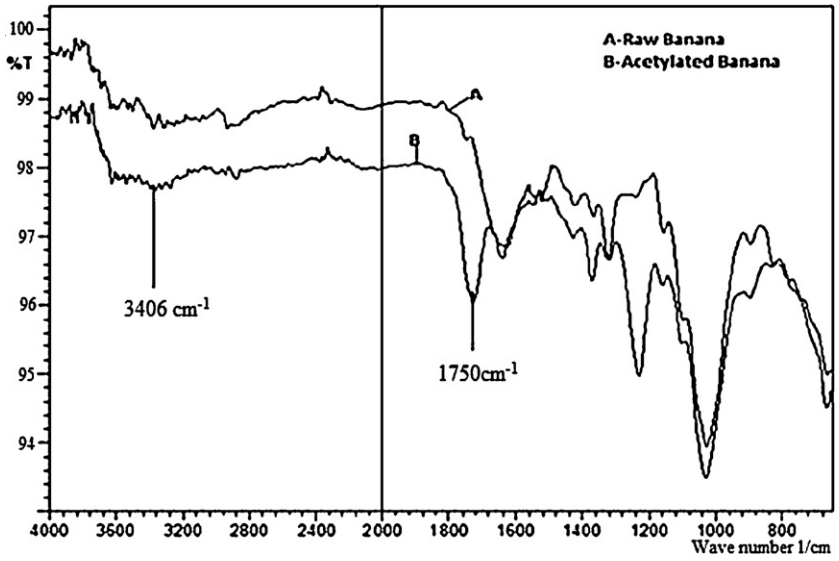

Fig. 1. FT-IR spectra of raw banana and acetylated banana fibre.

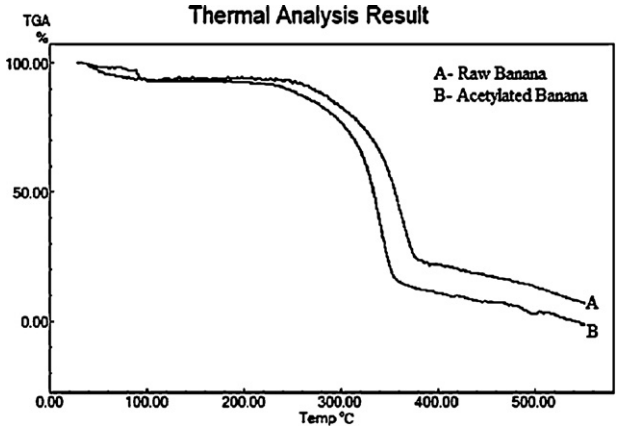

Fig. 2. TGA of raw and acetylated banana fibre.

for acetylated banana fibre at 350 °C. However beyond 350 °C, the weight loss was slowed down and finally at 550 °C it was found to be 92.56% for raw banana fibre and 99.11% for acetylated banana fibre respectively. This clearly indicates that the acetylated banana fibre showed relatively lower thermal stability as compared to raw banana fibre. The plausible reason could be that the disintegration of intermolecular interactions such as hydrogen bonds between polymer molecules during acetylation (Nwankwere et al., 2011).

4.7. SEM

In Fig. 3a it is observed that the untreated fibre surface is rough, exhibiting waxy and protruding parts. On acetylation reaction (Fig. 3b) the wax and cuticle on the surface are removed by the interaction with acetylating agent and surface becomes smoother. The fibrillation is also found to occur as the binding material is also removed and some micro pores appear in the treated fibres. Increased thickness, smooth surface, bulky groups clearly show the degree to which the internal 'channels' in the fibre open up and at the same time become straighter and confer greater rigidity

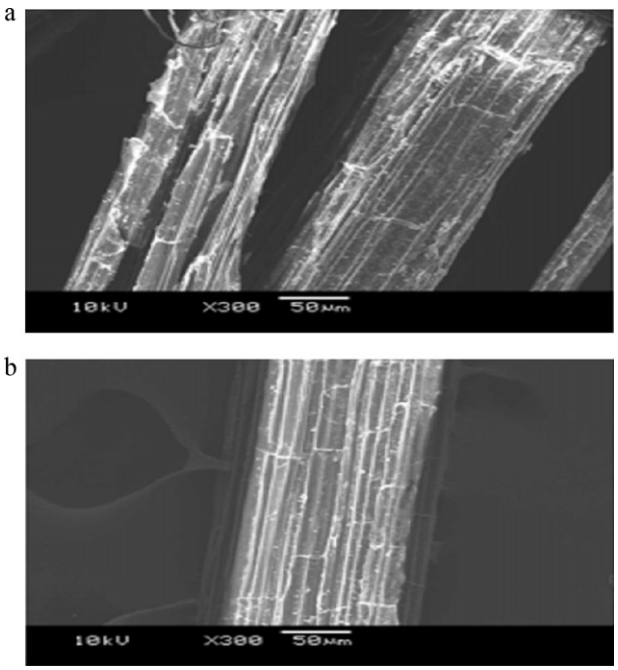

Fig. 3. Scanning electron micrograph of raw and acetylated banana. (a) SEM of raw banana fibre. (b) SEM of acetylated banana fibre.

upon the fibre. It is for this reason oleophilicity of the treated fibre increases.

4.8. Degree of acetylation and substitution

The sample which showed optimum level of WPG exhibited degree of acetylation (acetyl %) and substitution of the order of 14.25% and 0.69, respectively which confirmed that the banana fibre samples were successfully acetylated.

4.9. Oil absorption

Acetylation of banana fibre led to a substantial increase in acetyl groups. The modified fibre was, therefore, significantly hydrophobic and did not get wet with water. In this study, the oil absorption of the acetylated fibre was examined using machine oil whose viscosity was 1800 cP at 10 shear rate. The oil was suspended in water and modified fibre samples were introduced in it. The machine oil in water was immediately sorbed by the modified fibre. The capacity of oil sorption at room temperature of the acetylated banana samples (obtained by 1 h acetylation at 120 °C by using 2% NBS as a catalyst) was 18.12 g/g (refer Table 1) which in general, increased with the increment of WPG. The oil sorption capacity of raw banana fibre was 2.10 g/g and these values for modified banana samples were much higher than those for the synthetic sorbents (Choi & Cloud, 1992; Hill, Jones, Strickland, & Cetin, 1998).

4.10. Reusability of sorbents

The mechanical compression or squeezing method of recovering the oil from the sorbent material is a common, economical and practical one. Hence recovery of the oil from the sorbent material and also the feasibility of reusing the sorbents were studied. The results in Table 2 indicate that the acetylated banana fibre shows higher oil uptake in the first cycle and then its absorption capacity decreased significantly in the subsequent cycles. This may be due the collapsing of lumen during the mechanical squeezing (Hill et al., 1998) and masking of acetylated groups in banana fibre by mechanically held residual oil layer, even if squeezing was done which reduced the force of interaction between the fresh oil and previously used modified banana sorbent.

Table 2
Reusability of acetylated banana fibre.

	Oil sorbed (g of oil/g of fibre)	Oil remaining in fibre (g of oil/g of fibre)
First cycle	18.12	3.94
Second cycle	9.34	1.41
Third cycle	7.28	1.14

Table 3
Composition of raw and acetylated banana fibre.

Composition	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Raw banana fibre	57.64	29.05	13.30
Acetylated banana fibre, 13.89% WPG experimental values	50.61	33.81	15.54
Acetylated banana fibre (theoretically predicted)	(50.61)	(33.08)	(15.14)

4.11. Effect of fibre composition on acetylation

The influence of acetylation on the composition of the fibres is seen from Table 3 which indicates percentage composition of raw banana and modified banana in terms of cellulose, hemicellulose and lignin. The results clearly indicate that on acetylation, the overall percentage of cellulose was decreased; whereas hemicellulose and lignin which remain unaffected during the acetylation process, their relative percent composition obviously increased. The acetylated sample analysed here had shown 13.89 WPG upon acetylation of the raw banana fibre. Hence when such a sample was analysed for cellulose composition, naturally it is expected that there would be reduction in cellulose composition and thus cellulose in acetylated sample was experimentally found to be 50.61%, as compared to 57.64% in the unmodified sample. Theoretically speaking, taking into consideration of 13.89% weight gain, the modified sample should show cellulose content 50.61% $\{=(100/113.89) \times 57.64\}$. Similarly the other two components, namely hemicellulose and lignin should show increase in their relative proportion and theoretically they should show 33.08% $\{=(113.89/100) \times 29.05\}$ and 15.147% $\{=(113.89/1000) \times 13.30\}$, respectively. The experimental values for cellulose, hemicellulose and lignin thus are found to match with those theoretical values predicted within the acceptable limit.

Due to acetylation hydroxyl groups are replaced by bulkier acetyl groups resulting in increase in hydrophobicity and also decrease in H-bonding. It also gives rise to increase in accessibility or amorphous content. Both these factors thus cause enhancement in oleophilicity which has been depicted by these results.

5. Conclusion

The acetylation of the free hydroxyl groups in banana fibre with acetic anhydride without the use of solvent represents a suitable and effective method for the preparation of cellulose acetates, oil sorbents based on banana fibre having more hydrophobic characteristics. The WPG and oil sorption capacity increased with increments of reaction temperature and amount of catalyst used and the optimum WPG found was 13.89%. The thermal stability of modified fibre was found to be lower than that of the unmodified fibre. More importantly, it was found that the oil sorption capacities of the acetylated banana fibres were much greater (18.12 g/g of sorbent) than those of the synthetic sorbents such as

polypropylene fibre. It was also possible to reuse the acetylated banana fibre for oil absorption for at least three times. Low cost, high capacity, quick oil uptake and easy to desorb oil are the added advantages of the acetylated banana fibre. Thus, they could be used effectively to recover oil spilled in bodies such as in lakes, rivers, and oceans.

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