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RESPONSE SURFACE OPTIMISATION OF COTTON FIBRE ACETYLATION FOR OIL SORPTION USING CENTRAL COMPOSITE DESIGN

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ABSTRACT

Acetylation enhances the hydrophobicity of lignocellulosic biomasses by replacing their dominant hydrophilic hydroxyl groups with hydrophobic acetyl groups, thereby increasing their suitability for oil sorption. This modification typically results in weight gain due to the higher molecular mass of the introduced functional groups. This study employed the Central Composite Design (CCD) Response Surface Methodology (RSM) to optimise cotton fibre acetylation to enhance its weight percent gain (WPG) and improve its suitability for oil sorption. Reaction time, temperature, and the fibre mass-to-volume ratio of acetic anhydride were investigated, with WPG as the response parameter. Among the 20 experimental runs, the highest weight gain of 4.782% was achieved at 2 hours, 137°C, and a mass-to-volume ratio of 0.011 (0.550 g/50 ml). However, optimisation of the experimental conditions yielded a predicted WPG of 4.950% at 2.584 hours, 129.964°C, and 0.258 g of fibre per 50 ml of acetic anhydride. Validation of the predicted optimum response resulted in an experimental WPG of 4.896%, closely matching the predicted value of 4.950%. The minimal deviation of 0.054 demonstrated the model's high accuracy and reliability. Additionally, the desirability score of 1.000 indicated that the optimised conditions perfectly aligned with the study's objectives. Successful acetylation was confirmed through FTIR analysis, which provided evidence of the introduction and enhancement of peaks associated with acetylated products.

***Keywords**: Cotton, Acetylation, Response Surface and Optimisation.*

1. INTRODUCTION

Cotton fibre is a promising natural alternative to synthetic sorbents for oil spill cleanup (Silva, *et al*., 2023; Nguyen, *et al*., 2023; Luo *et al*., 2013; Adebajo and Frost, 2004; Agida, *et al*., 2017). They are primarily composed of cellulose (82-96%), hemicellulose (2-6.4%), lignin (0-5%), and pectin (<1-7%) (Tu, *et al*., 2024; Bahlool and Kamel 2023), cotton exhibits a robust cellular structure due to rigid cellulose chains and extensive intermolecular and intramolecular hydrogen-bonding (Fang, *et al*., 2024; Hsieh, 2007). This structure enhances its durability compared to fibre-like kapok. However, cotton's hydrophilic nature limits its oil sorption capacity, necessitating modifications by substitution of hydroxyl functional group with hydrophobic moieties. Previous studies of cotton, kapok, rice husk, corn cob, banana and orange peel and lignocellulose biomass in their natural form show low oil sorption capacity compared to the synthetic sorbent (Olawale and Saidu, 2010; Saidu 2011; Olawale *et al*., 2022a; 2022b). **Acetylation is one of the methods for enhancing the oil-absorbing properties of lignocellulosic biomass,** transforming it into a hydrophobic, oil-absorbing material suitable for oil spill cleanup. This process involves replacing the hydrophilic hydroxyl groups in the biomass with hydrophobic acetyl groups, as represented in Equation 1. Since acetyl groups have higher molecular mass than the hydroxyl groups, acetylation typically increases the biomass weight, hence the use of weight gain to ascertain the level of acetylation (Heo *et al*., 2024; Teli and Terega, 2022)

$$\underset{\text{Lignocellulose}}{LC-OH} + \underset{\text{Acetic anhydride}}{R_2(C=O)_2O} \rightarrow \underset{\text{Acetylated LC}}{LC-(C=O)R} + \underset{\text{Acetic acid}}{RCOOH} \quad (1)$$

Acetylation of some lignocellulose biomass for oil spill clean-up has been reported in the literature (Fasanya, *et al*., 2020; Mahmoud, 2020; Nwadiogbu, *et al*., 2016; Onwuka, *et al*., 2019; Teli and Valia, 2013a, 2013b; Chung, *et al*., 2011; Sun, *et al*., 2004; Agida, *et al*., 2017; Adebayo and Frost 2003; Luo, *et al*., 2013). Whereas, previous studies employed the one-factor-at-a-time (OFAT) approach, which is limited in capturing factors interaction and identifying optimum conditions, this study aimed at utilising the response surface methodology (RSM) central composite design CCD, considered more robust, for process optimisation, accounting for factors interactions, optimal experimental conditions, deeper

understanding of process conditions to enhance performance. Luo *et al*., 2013 have successfully proposed the kinetic model for cotton linter pulp acetylation catalysed by sulfuric acid. Adebayo and Frost (2004) reports have shown successful acetylation of cotton fibre by reaction with acetic anhydride in the presence of DMAP catalyst as indicated by both FTIR and NMR. However, acetylating temperature, time and ratio of the mass of the biomass to the volume of the acetylating agent are factors which could significantly affect the extent of acetylation based on a particular biomass structural configuration. The combined effect of these factors on the weight per cent gain and extent of acetylation using response surface methodology (RSM) are not deeply established in the previous studies. Hence, this study is aimed at enhancing the acetylation of cotton fibre for crude oil sorption using design expert software response surface methodology, having been established as a reliable tool for process optimisation. In addition, to establish the significance of acetylating temperature, time and ratio of cotton fibre to the volume of the acetylating agent on the acetylation of cotton fibre.

2. MATERIALS AND METHODS

2.1 Material Collection and Preparation

The cotton fibre (SAMCOT-9 variety) was sourced from the Fibre Unit of the Institute of Agricultural Research, Ahmadu Bello University Zaria, the seed was harvested in the 2022/2023 season and manually ginned to remove the cotton fibres, and debris was carefully removed from the fibre, it was air dried and subsequently oven dried at 60 ^{0}C for 8 hours to remove moisture content.

2.2 Experimental Design

Design Expert Version 13 software (Stat-Ease, Inc.) was employed in the study using the Central Composite Design (CCD) as an optimisation tool within Response Surface Methodology (RSM) to investigate how the variables influence the acetylation process, based on its efficiency in model complex relationships between variables (Mourabet *et al*, 2017; Bayuo, 2020). It was used to develop predictive equations and identify optimal process conditions for the acetylation process. CCD can be implemented as circumscribed (CCC), inscribed (CCI), or face-centred (CCF) designs. CCC was selected for this study due to its larger experimental region compared to CCI and CCF (Bhattacharya, 2021).

To enhance the cotton fibre acetylation, three independent variables were considered: reaction time, temperature, and sorbent mass/acetic anhydride ratio. Prior research conducted by Luo et al. (2013) investigated the acetylation of cotton linter pulp utilising the One Factor at a Time (OFAT) approach, specifically focusing on reaction time, temperature, and mass-to-volume ratio at 0.5-2 hours, 30-45 °C, and 2g/80ml (0.025) respectively. Similarly, Mahmoud (2020) investigated the acetylation of flax fibre, which was undertaken precisely at 1 hour and 65 °C with a mass-to-volume ratio of 5g/100ml (0.05). While, Fasanya et al. (2020) examined the reaction time, temperature, and mass-to-volume ratio for *Sansevieria liberica* acetylation, specifically at 3 hours, 70 °C, and 2g/60ml (0.033) respectively.

Based on the previous studies (Fasanya et al. 2020; Mahmoud 2020; Luo et al., 2013), a wider range of reaction time, temperature, and mass-to-volume ratio were selected for this study as shown in Table 1, using the response surface methodology due to its efficacy in elucidating the significance of process parameters, their interactions, and its capacity to ascertain optimal conditions.

Table 1: Experimental Build Information

Factor	Name	Unit	Min	Max	Coded Low	Coded High	Mean	Std. Dev
A	Time	hr	0.2000	3.80	-1 ↔ 0.50	+1 ↔ 3.50	2.00	1.14
B	Temperature	^{0}C	53.00	137.00	-1 ↔ 60.00	+1 ↔ 130.00	95.00	26.49
C	Solid/ Liquid	g/50 ml	0.0100	1.09	-1 ↔ 0.10	+1 ↔ 1.00	0.5500	0.3405

2.2 Fibre acetylation

A mixture of the oven-dried fibre and acetic anhydride in the range of experimental variables (Table 1) with NBS catalyst (2 % of the acetic anhydride) was heated between 53 – 137 ^{0}C under reflux in a 250 ml round-bottom flask fitted with a condenser for a duration of 0.2 hour – 3.8 hours. At the end of the reaction time, the hot reagent was decanted off and the acetylated cotton was thoroughly washed with ethanol and acetone to remove the unreacted acetic anhydride and the acetic acid product.

Furthermore, drying of the acetylated fibre in an oven at 60 ^{0}C for 16 hours, after which the weight gain was determined by Shimadzu ATX224 digital weighing balance (0.0001 g) and subsequently stored in a desiccator at room temperature as carried out in previous studies (Adebayo and Fost, 2004; Sun, *et al.,* 2004; Onwuka *et al.*, 2018; Nwadiogbu *et al.,* 2016; Fasanya, *et al*., 2020).

2.3 Fourier transform infrared (FTIR) Analysis

The FTIR was performed using Shimadzu-8400S Fourier transform infrared (FTIR) spectrophotometer within the spectra range of 4000–500 cm^{-1}. The IR spectra were analysed using spectroscopic software Win-IR Pro (Version 3.0). 2 mg of fibre was mashed, mixed with 200

mg KBr, and then pressed into 1 mm-thick disks, the sample was placed in the FTIR spectrometer and its beam directed at the sample to measure how much of the beam and at which frequencies the sample absorbs the infrared light to identify the molecular identity of the fibres.

2.4 Optimisation of Experimental Conditions for WPG Enhancement and its Validation

The desirability function was used to predict the best conditions, maximizing the response variable while considering desired constraints. The selection of optima conditions from CCD was based on the highest desirability of 1 to maximum predicted response (highest fibre weight gain). The optimised conditions were applied in a real experiment and the actual response measured and compared with the predicted response followed by error analysis. The percentage variation between predicted and actual values was calculated to assess model accuracy as shown in Equation 2.

$$\% \text{ Error} = \frac{\text{Predicted} - \text{Experimental}}{\text{Predicted}} \times 100 \qquad (2)$$

3. RESULTS AND DISCUSSION

3.1 Response surface analysis of cotton weight per cent gain after acetylation

The introduction of acetyl moieties into the cotton fibre consequently results in to increase in the mass of the acetylated fibre due to the higher molecular mass of the hydrophobic acetyl (Heo *et al*., 2024). However, the weight gain varied correspondingly under different combinations of reaction time, temperature and fibre-to-acetic anhydride ratio (solid/liquid) as shown in Table 1 indicating the influence of the investigated parameters on the responses.

Table 1: CCD Experimental Design Template and Corresponding Experimental Responses

	Factor 1	Factor 2	Factor 3	Response 1
Run	A: Time	B: Temperature	C: Solid/Liquid	WPG
	h	^{0}C	g/50 ml	%
1	2	53.0	0.55	0.164
2	0.2	95.0	0.55	0.218
3	2	95.0	0.55	3.673
4	2	95.0	0.55	3.691
5	3.5	130.0	0.10	4.700
6	0.5	60.0	1.00	0.120
7	2	95.0	0.55	3.201
8	2	95.0	0.55	3.691
9	0.5	130.0	1.00	1.350
10	2	95.0	0.01	4.000
11	2	137.0	0.55	4.782
12	2	95.0	1.09	3.679
13	3.5	60.0	1.00	0.430
14	3.5	60.0	0.10	0.400
15	0.5	130.0	0.10	1.500
16	2	95.0	0.55	3.891
17	2	95.0	0.55	3.691
18	3.8	95.0	0.55	3.255
19	3.5	130.0	1.00	4.750
20	0.5	60.0	0.10	0.100

In Table 2, analysis of the experimental data based on weight per cent gain shows a preferable fit to the quadratic model compared to the linear and two-factor interaction (2FI) with cubic models being aliased within the range of the investigated parameters. The preference for the quadratic model over the cubic model is attributed to the significance of only two out of the three investigated factors. While the factors' interaction may be described by the cubic model, the quadratic may offer better fits where all factors are not significant within the

range of investigations (Iro, *et al*., 2024 Kousha *et al*., 2015; Das, 2017). Therefore, the quadratic model offers a good balance between capturing essential response surface features and keeping the model interpretable and manageable. The appropriateness of the model is further confirmed by the analysis of variance (Table 3), which shows a significant model with an F-value of 57.10 and a p-value less than 0.0001. In CCD analysis of variance, a p-value greater than 0.05 and an F-value less than 0.05 typically confirm the significance of the model and its terms (Oladipo, *et al*., 2018; Eletta, *et al*., 2016). The analysis of variance reveals that the most significant model terms are acetylating temperature (A) and time (B), followed by the interacting terms AB, A^2 and B^2. However, the data suggests that the fibre-to-acetylating agent ratio (C) is the least significant independent variable, particularly at elevated temperatures. Implying that solid-to-liquid ratio is less critical compared to other reaction parameters in the acetylation of cotton.

Table 2: WPG Fit Summary for Cotton Fibre Acetylation

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	
Linear	0.0015	0.0004	0.5352	0.3309	
2FI	0.4168	0.0003	0.5367	-0.4932	
Quadratic	**< 0.0001**	**0.1049**	**0.9637**	**0.8791**	**Suggested**
Cubic	0.0402	0.9286	0.9861	0.9896	Aliased

While solid-to-liquid ratio generally affects most processes as a component moves from the solid to the liquid phase and vice versa for efficient mass transfer, however, the significance of the solid-to-liquid ratio may depend on the specific process with other factors having a stronger influence on the response variable as in this process. Furthermore, unlike most reactions where reactants are fully dissolved, cotton acetylation is a heterogeneous process with the cotton and acetic anhydride/acetylating agent in different phases and reaction occurring at the interface between the solid cotton fibres and the liquid acetic anhydride, thereby suppressing the solid to liquid ratio effects. In addition, the acetylation was carried out in a solvent-free state with a high concentration of acetylating agent in the liquid phase may have enhanced effective collisions with cellulose molecules at high temperature, thereby limiting the solid-to-liquid ratio effects on the responses compared to reaction time and temperature which directly influence the rate and extent of acetylation within the cellulose fibre. However, minimum amount of liquid is still necessary to ensure the proper distribution of reactants and efficient mass transfer throughout the reaction mixture while maintaining the fibrous structure.

Table 3: Weight Percent Gain Analysis of Variance (ANOVA)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	59.25	9	6.58	57.10	< 0.0001	significant
A-Time	10.83	1	10.83	93.93	< 0.0001	
B-Temperature	25.92	1	25.92	224.79	< 0.0001	
C-Solid/Liquid	0.0174	1	0.0174	0.1510	0.7057	
AB	4.49	1	4.49	38.90	< 0.0001	
AC	0.0055	1	0.0055	0.0478	0.8313	
BC	0.0028	1	0.0028	0.0244	0.8790	
A^2	8.95	1	8.95	77.65	< 0.0001	
B^2	3.34	1	3.34	28.96	0.0003	
C^2	0.1114	1	0.1114	0.9660	0.3489	
Residual	1.15	10	0.1153			
Lack of Fit	0.8883	5	0.1777	3.36	0.1049	not significant
Pure Error	0.2646	5	0.0529			
Cor Total	60.40	19				

To further verify the model fitness, a residual error (pure error) or variability of 0.2646 in Table 3 was obtained in the weight per cent gain response analysis, this represents an unexplained variability by the experimental factors, value this low indicates higher precision in the experiment. Signifying that the model is a good fit for the data with most of the variability in the response variable explained by the experimental factors (Kousha, *et al*., 2015). In addition, a value of 0.2646 suggests the chosen model captures a very high proportion of the variation observed in the response variable. Similarly, a mean square error (MSE) of 0.0529, which is an average of the squared pure errors, representing the average variability in the response variable that is not explained by the model was obtained, also confirming the model fitness. The lack of fit (f-value) of 0.1049 also confirms the significance of

the chosen model, implying that the lack of fit is not significant relative to the pure error. In addition, the adjusted R^2 of 0.9637 is in reasonable agreement with the predicted R^2 of 0.8791 with a difference of 0.0846, which is less than 0.2 as shown in Table 4. An adequate precision of 21.5000 which is greater than 4 was obtained, this affirms the ability of the model to navigate the design space.

Table 4: Weight Percent Gain Fit Statistics

Std. Dev.	0.3395	R^2	0.9809
Mean	2.56	Adjusted R^2	0.9637
C.V. %	13.24	Predicted R^2	0.8791
		Adeq Precision	21.5000

$$WPG = 3.63584 + 0.997647A + 1.54335B - 0.04C + 0.74875AB + 0.02625AC - 0.01875BC - 1.31387A^2 - 0.802413B^2 + 0.146545C^2 \quad (2)$$

Equation 2 is the predictive empirical model expression in terms of the coded factors for the weight per cent gain response which indicates the relative impact of the factors by comparing the factor coefficients. In Figure 1, the closeness of the experimental value to the predicted value indicates that the responses obtained from the experimental results properly fit within an acceptable variance range when compared to the predicted values from the empirical model, this is an indication of the high R^2 value of 0.9809 obtained in Table 4, as seen in Figure 1 with most of the points distributed relatively near the 45-degree diagonal line with no much outliers. This points to the adequacy of the model for predicting the response parameter within the range of the investigated variables.

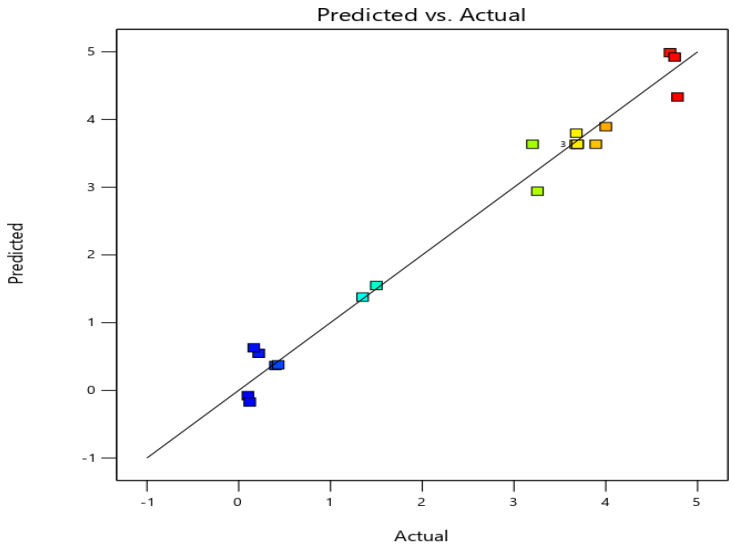

Figure 1: Correlation Between Predicted and WPG Actual Experimental Data

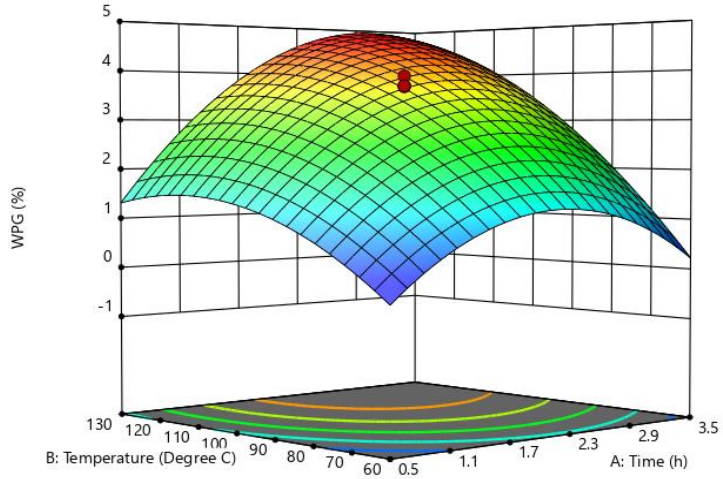

Figure 2: 3D Response Surface Plot for the Variables Effects on Cotton Weight Percent Gain

Figure 2 represents the resultant effect of the independent variables on the weight per cent gain response, indicating that an increase in acetylating temperature and reaction time leads to an increase in the cotton fibre weight per cent gain to an optimum point of about 4.782 weight gain. However, the weight gain was not significant at a reaction time of 0.5 minutes and temperature of 60 ^{0}C, suggesting the requirement of a higher reaction time and temperature for successful acetylation. This is in agreement with Sun *et al*., 2004 and Chung et *al*., 2011 which reported visible acetylation after 1 hour and above 100^{0}C for Sugarcane bagasse. However, for cotton, acetylation became more substantial at 2 hours and a temperature of 95 ^{0}C with an increase in weight per cent gain to above 3.60.

3.2 FT-IR results analysis

Using FTIR spectroscopy, the successful acetylation of the fibre was verified based on functional group transformation after acetylation, to identify the absence or presence of peaks associated with acetylated products. Typically, these peaks are visible at 1745 cm^{-1}, 1376 cm^{-1} and 1234 cm^{-1} representing carbonyl stretching of ester, C-H in -O(C=O)-CH_3 and C-O peak of acetyl group. The FT-IR spectra of the acetylated fibre are shown in Figure 3-8. In Figure 3 the spectral of the cotton fibres acetylated at 0.5 hour, 60 ^{0}C, shows no significant transformation at fibre to the volume of acetic anhydride ratio of 0.1g/50ml and 1g/50ml. However, at 0.20 hour, 95^0 (Figure 4) and 0.5 hour, 130 ^{0}C (Figure 5) a more visible appearance of peak at 1745 cm^{-1} was observed, similarly at 2 hours and 95 ^{0}C (Figure 6) the carbonyl stretching of ester at 1745 cm^{-1} became distinctly visible, implying the dependence of successful acetylation on reaction time and temperature and not singly on each of the factor, this is also a confirmation of the significant of these factors in the weight gain analysis of variance (Table 3) and corresponding model equation (Equation 2).

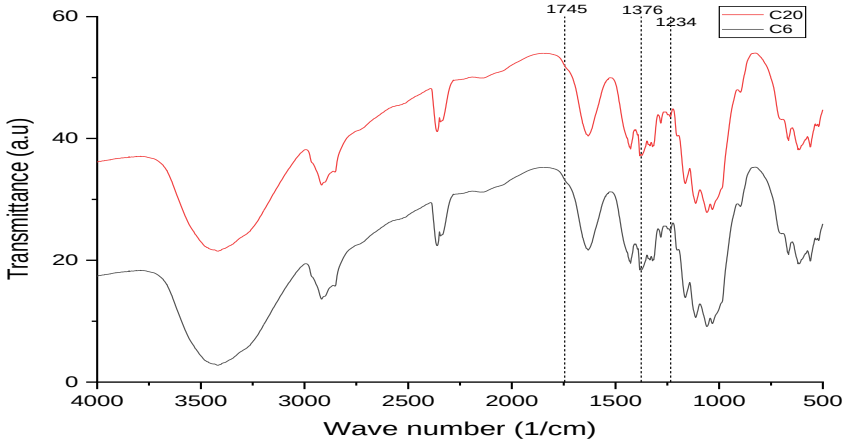

Figure 3: FT-IR spectral of acetylated cotton fibre at 0.5 hour, 60 ^{0}C, 1 g cotton fibre/50ml acetic anhydride (C_{20}) and 0.5 hour, 60 ^{0}C, 0.1 g cotton fibre/50ml acetic anhydride (C_6)

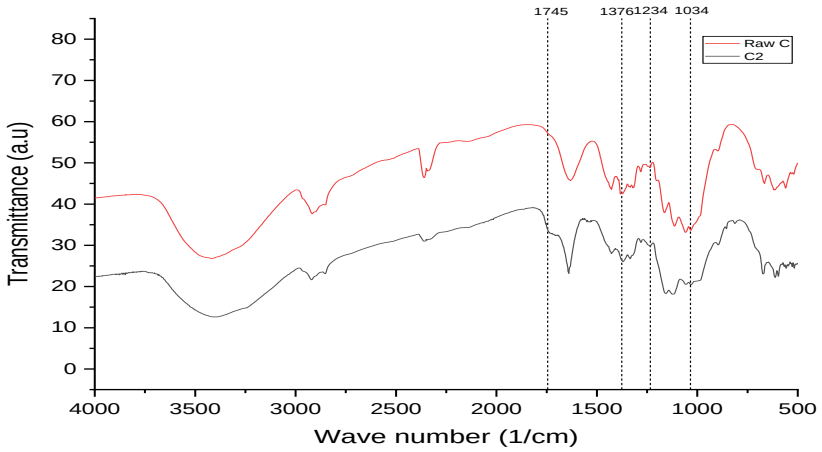

Figure 4: FT-IR spectral for acetylated cotton fibre at 0.2 hour, 95 ^{0}C, 0.55 g cotton fibre/50ml acetic anhydride

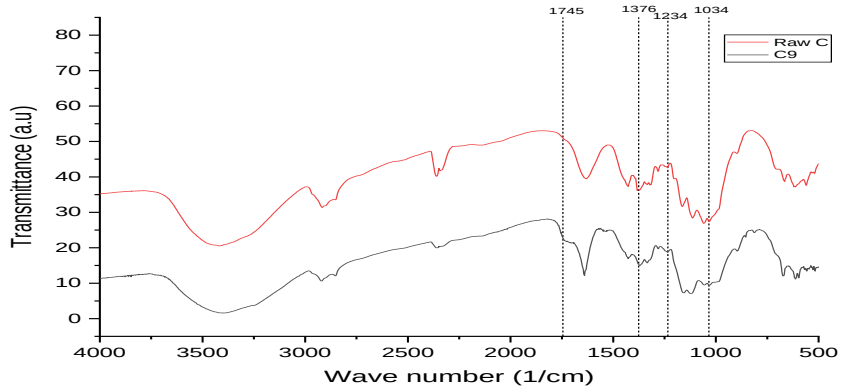

Figure 5: FT-IR spectral for acetylated cotton fibre at 0.5 hour, 130^{0}C, 1 g cotton fibre/50ml acetic anhydride

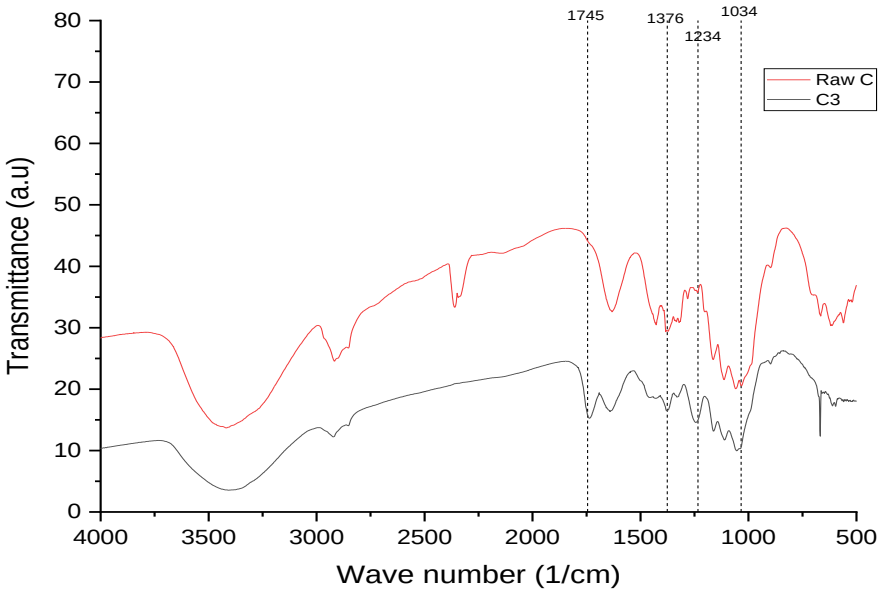

Figure 6: FT-IR spectral of acetylated cotton fibre at 2 hour, 95^{0}C, 0.55 g cotton fibre/50ml acetic anhydride

A comparison of the FT-IR spectral of unmodified cotton fibre (Raw C) with acetylated cotton fibre at 3.5 hour, 130 ^{0}C, 1 g cotton fibre/50ml acetic anhydride and 3.5 hour, 130 ^{0}C, 0.1 g cotton fibre/50ml acetic anhydride (Figure 7) shows very significant transformation with visible appearance of peaks at 1745 cm^{-1}, 1376 cm^{-1} and 1234 cm^{-1} representing carbonyl stretching of ester, C-H in -O(C=O)-CH_3 and C-O peak of acetyl group respectively. However, the typical characteristic peaks associated with raw cotton fibres remain present at 1376 cm^{-1}, 2922 cm^{-1} and 3420 cm^{-1}, representing the bending vibration of C–H, the stretching vibration of C-H, and the stretching vibration of O-H bond respectively.

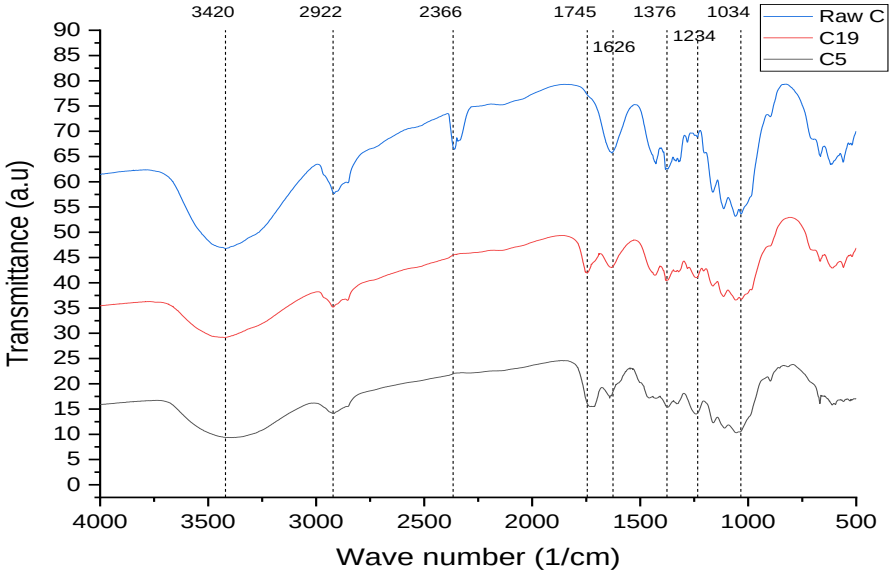

Figure 7: FT-IR spectral of unmodified cotton fibre (Raw C) and acetylated cotton fibre at 3.5 hour, 130 ^{0}C, 1 g cotton fibre/50ml acetic anhydride (C_{19}) and at 3.5 hour, 130 ^{0}C, 0.1 g cotton fibre/50ml anhydride (C_5)

Cotton fibre is about 90 % cellulose and typically hydrophilic due to the predominance of the O-H band associated with highly cellulosic biomass. The decrease in the intensity of the O-H stretching at 3420 cm^{-1} in the modified cotton fibre is evidence of the successful replacement of the hydrophilic O-H bond with the enhancement of the hydrophobic acetyl groups (Chung *et al*., 2011); this was further confirmed by the decrease in the intensity of the H-O-H bending peak at 1626 cm^{-1}(Onwuka et al., 2016). The strong absorbance band at 1034.04 cm^{-1} is ascribed to the C–O and O–H stretching vibration of the polysaccharide in cellulose (Olawale *et al*., 2020a). The FTIR bands of the raw and modified cotton fibres also conform with previous reports on cotton fibre acetylation (Lv, *et al*., 2018; Bello *et al*., 2016; Adebajo & Frost, 2004). From the experimental data, a maximum weight gain of 4.782% was observed under conditions of 137 °C for 2 hours with a fibre-to-acetic anhydride ratio of 0.55 g/50 ml, this finding aligns with the FT-IR spectra presented in Figure 8. A comparison of the unmodified cotton (Raw C) spectrum to that obtained under these optimal conditions reveals a significant reduction in the hydroxyl band at 3420 cm^{-1}, indicating extensive O-H substitution. No peak was observed in the region of 1840–1760 cm^{-1} in the spectra of all the acetylated cotton samples indicating that the acetylated products are free of unreacted acetic anhydride (Sun *et al*., 2004).

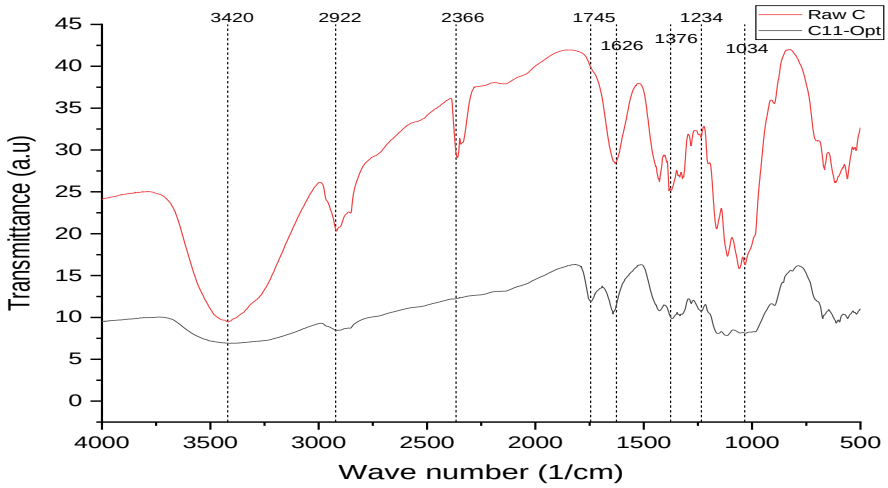

Figure 8: Comparison of the FT-IR spectral of the unmodified cotton (Raw C) and optimum extent of acetylation cotton (2 hours, 137 ^{0}C, 0.55 g cotton fibre/50ml acetic anhydride) (C_{11})

Similarly, the absence of a peak at 1700 cm^{-1} for a carboxylic group in all the spectra of the acetylated samples also indicates that the acetylated products are free of the acetic acid by-product (Chung *et al*., 2011; Sun *et al*., 2004), these confirms the thorough rinsing of the acetylated products with acetone and ethanol after acetylation as shown in Plate 4.1, in addition the reliability of weight gain as basis for establishing extent of acetylation.

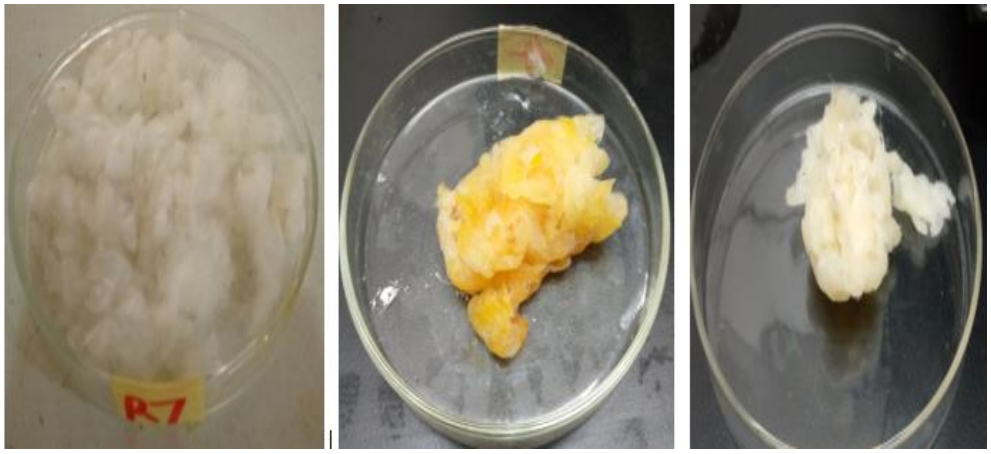

Plate 1 (a): Raw cotton fibre (b): Fibre after acetylation (c) Rinsed acetylated product

3.3 Optimisation of Acetylated Cotton Fibre Weight Gain

Optimizing experimental conditions helps to achieve the desired optimum response in response surface methodology (Uzochukwu et al., 2024). A study by Adebayo and Frost (2004) reported weight gain as a reliable index for determining the extent of acetylation in cotton fibre. Therefore, an attempt was made to enhance cotton fibre weight gain by considering experimental conditions within the range of investigated parameters in the optimisation constraints, as shown in Table 5. Several optimised solutions were generated, as shown in Table 6. Solution Number 1 presents the predicted optimal conditions of 2.584 hours, 129.964°C, and 0.258 g of fibre per 50 mL of acetic anhydride.

Table 5: Weight Gain Optimisation Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A: Time	is in range	0.5	3.5	1	1	3
B: Temperature	is in range	60	130	1	1	3
C: Solid/Liquid	is in range	0.1	1	1	1	3

WPG	maximize	0.1	4.782	1	1	3
Oil Sorption Capacity	maximize	32.5	47.01	1	1	3

Table 6: Weight Gain Optimisation Solutions

Number	Time	Temperature	Solid/Liquid	WPG	Desirability	
1	**2.584**	**129.964**	**0.258**	**4.950**	**1.000**	**Selected**
2	3.261	129.137	0.530	4.904	1.000	
3	2.605	129.403	0.118	5.044	1.000	
4	3.500	130.000	0.100	4.988	1.000	
5	2.611	128.856	0.293	4.936	1.000	

Table 7: Validation of Optimal Conditions for Weight Gain

Number	Time (h)	Temperature ^{0}C	Solid/Liquid (g/50ml)	WPG	Desirability
Optima (Predicted)	2.584	129.964	0.258	4.950	1.000
Validated	2.584	130	0.258	4.896	1.000

An actual experiment was conducted using the optimized (predicted) conditions from Solution Number 1, with the temperature rounded to 130°C. The experimental result yielded a WPG of 4.896 as shown in Table 7 which closely matched the predicted WPG of 4.950. The difference between the predicted and actual results was only 1.09 %, demonstrating the model's high accuracy and reliability. The desirability score of 1.000 indicates that the optimised conditions perfectly align with the study's objectives, confirming that the selected conditions are ideal for maximizing cotton fibre weight gain. The small error margin in the validation results further supports the model's reliability.

3. CONCLUSION

An examination of cotton fibre acetylation under varying reaction conditions— temperature, time, and fibre-to-acetic anhydride ratio revealed that reaction time and temperature are critical factors influencing the process. This finding was validated by FT-IR analysis. Acetylation was limited at 2 hours and 53 °C, 0.2 hours and 95 °C, and 0.5 hours and 130 °C, as evidenced by weight gain and FT-IR spectra. However, acetylation significantly improved at 2 hours and 95 °C, with minimal influence from the fibre-to-acetic anhydride ratio. Under the condition of 2 hours, 137 °C, and a fibre-to-acetic anhydride ratio of 0.011 (0.550 g/50 mL) the highest WPG was obtained amongst the 20 experimental runs. However, the CCD-based optimisation approach successfully identified reaction time of 2.584 hours, 129.964°C, and 0.258 g of fibre per 50 ml of acetic anhydride as the optima conditions with predicted WPG value of 4.950 %. Validation of the predicted optimum response resulted in an experimental WPG of 4.896 %, closely matching the predicted value of 4.950 %. The minimal variation in the validation results confirmed the effectiveness of the optimisation model.

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