Lafia Journal of Scientific & Industrial Research (LJSIR), Vol. 1(1&2), 2023
e-ISSN:... p-ISSN: 3026 – 9288 pages: 12-17
https://lafiascijournals.org.ng/index.php/ljsir/index ⇒ Published by the Faculty of Science,
Federal University of Lafia, Nasarawa State, Nigeria

Comparative Evaluation of Analytical Techniques Used in Estimating Acetylation of Lignocellulosic Materials

Jude Chinedu Onwuka[1]*, Imam Akeyede[2] & Enebi Estella Jasper[3]
[1]Department of Science Laboratory Technology, Federal University of Lafia, PMB 146, Nasarawa State – Nigeria
[2]Department of Mathematics and Statistics, Federal University of Lafia, PMB 146, Nasarawa State – Nigeria
[3]Department of Chemical Sciences, Dennis Osadebay University, Asaba, Delta State – Nigeria

Abstract

The techniques commonly used in estimating extent of acetylation are based on the principle of substituting hydroxyl groups with acetyl groups. In this study, three lignocellulosic materials were modified using solvent free method of acetylation with NBS (N – bromosuccinimide) as catalyst. The extent of modification of these materials were estimated using three techniques – weight percent gain (WPG), extent of acetylation (R) and degree of substitution (DS). Six (6) factors were considered in the acetylation of the lignocellulosic materials. Equality of variance – covariance matrices of the techniques across the factors in all the materials were tested with Box's M test. The performance and response of the techniques to variation of the factors studied were compared statistically using multivariate analysis (MANOVA) and Duncan multiple range test. MANOVA results showed no statistical difference on the response of the techniques towards variation of the factors studied in acetylating these materials. However, it also showed that there was significant difference on the performance of the techniques used in estimating extent of acetylation. Duncan multiple range test analysis indicated that WPG performed best in estimating extent of acetylation. Thus, any of the techniques can be used to estimate extent of acetylation satisfactorily.

Keywords: Weight percent gain, degree of substitution, Box's M, multivariate analysis, Duncan analysis, acetylation

Article History
Submitted
July 31, 2023
Revised
October 13, 2023
First Published Online
October 31, 2023

Corresponding author
J. C. Onwuka ✉
emperor20062003@yahoo.com

doi.org/10.62050/ljsir2023.v1n1.265

Introduction

Acetylation is one of the most commonly used chemical modification methods. This type of modification is obtained by esterification of cellulose with acetic anhydride, vinyl acetate or acetic acid [1]. In acetylation reactions, the hydroxyl groups on the cellulose are substituted with acetyl groups. Thus, the hydrogen bonding density between hydroxyl groups is reduced while the water – repelling characteristics of the fiber increase. Therefore, acetylation changes the surface of fibers from hydrophilic to hydrophobic by increasing the acetyl contents in the polymers while decreasing their –OH groups. As a result, acetylation provides a suitable method for producing cellulose acetates effectively used as reinforcement in polymer composites and oil removal from aqueous solution. Naturally, cellulose exist as lignocellulose because in its natural form it contains lignin and hemicellulose whose hydroxyl groups has different reactivity and thus, interferes with the extent of acetylating the lignocellulose. No two natural occurring lignocellulose contain the same amount of lignin and hemicellulose.

Weight percent gain (WPG), extent of acetylation (R) and degree of substitution (DS) techniques are used in the estimation of level of acetylation. These techniques are based on the principle of substituting hydroxyl groups with acetyl groups. WPG is a gravimetric technique which is based on the amount of weight added to the cellulose surface due to attachment of acetyl group which has higher molecular weight than the hydroxyl group. This technique has raised some accuracy doubt because of its inability to account for the exact weight gain due to loss of small sizes of sample particles during acetylation process. DS is a titrimetric technique based on the quantitative determination of the amount of –OH groups before and after acetylation. Questions have been raised on the accuracy of this technique in the determination of acetylation level in natural cellulose. This is because considering the difference in the reactivity of hydroxyl groups of lignin, hemicellulose and holocellulose, it will be difficult for some of the hydroxyl groups to be free for determination at the same reaction condition. The extent of acetylation (R) technique is an FTIR technique used in determining level of acetylation by calculating the ratio R between the intensity of the acetyl C=O stretching of ester at 1740 – 1745 cm^{-1} and the intensity of C–O stretching vibration of the cellulose backbone at about 1020-1040 cm^{-1} [2]. This technique is yet to raise any criticism or doubt since it apparently took care of the interferences by the different reactivity of lignin and hemicellulose hydroxyl groups.

Multivariate analysis of variance (MANOVA) is an extension of analysis of variance (ANOVA) applied to situations where there are two or more dependent variables [3]. In MANOVA, the number of response variables is increased to two or more. The hypothesis concerns a comparison of vectors of group means. When we run MANOVA in statistical package for social sciences (SPSS), we are presented with several lines of multivariate outcomes.

Each line reports potentially different significance, so it is important we select the correct one. There are four options: Pillai's Trace, Wilk's Lambda, Hotelling's Trace and Roy's Largest Root. Several factors determine which of the aforementioned four options, we can select. Hotelling's Trace should be used only when the independent variables are represented by two groups. It is not as powerful as some of the alternative choices. Wilk's Lambda is used when independent variables have more than two groups. Pillai's Trace and Roy's Largest Root can be used with any number of independent variables [4].

Post hoc procedures are often necessary after the null hypothesis is rejected in an ANOVA [5] or a MANOVA [3]. This is because the null hypotheses for these procedures often do not provide researchers with all the information that they desire [6, 7]. Tukey's test and Duncan's multiple-range test are two of the procedures that can be used and are found in most statistical packages [8]. Duncan's Multiple – range test is a procedure based on the comparison of the range of a subset of the sample means with a calculated least significant range. This least significant range increases with the number of sample means in the subset. If the range of the subset exceeds the least significant range, then the population means can be considered significantly different. It is a sequential test and so the subset with the largest range is compared first, followed by smaller subsets. Once a range is found not to be significant, no further subsets of this group are tested [9].

The purpose of this research is to statistically compare the use of three different techniques in estimating level of acetylation in three different natural fibers that are of different composition of lignin and hemicellulose. This is to determine the sensitivity of each technique to the variation of factors affecting acetylation and the performance of each technique in estimating extent of acetylation.

Materials and Methods

Sample collection and preparation

The sample materials; Oil Palm Empty Fruit Bunch (OPEFB) and Cocoa Pods (CP), were obtained from a local farm at Anambra State while Pride of Barbados Pods (POBPs) was collected from the environment of National Research Institute for Chemical Technology (NARICT), Zaria. The sample materials were cut and ground in a mortar, then, thoroughly washed with distilled water to remove foreign materials and water-soluble components. This allows the sample materials to maintain balance. The washed sample materials were allowed to dry properly in sunlight for 12 h and then left to dry at 65^{o}C in the oven to a constant weight.

After drying, the sample materials were sieved with laboratory sieves to obtain homogenous particle sizes using the BS410/1986 laboratory test sieve. A mechanical sieve shaker was used to separate the sample materials into the desired particle size (i.e., 425 – 625 µm).

Acetylation of the lignocellulosic materials

The acetylation of the lignocellulosics under mild conditions, in the presence of N – bromosuccinimide (NBS), using acetic anhydride were carried out using the Sun *et al.* [10] method of acetylation in a solvent free system.

A specific amount (3 g) of sample was placed in a 250 mL conical flask containing 60 mL of acetic anhydride and 0.6 g (1% of the solvent) N – bromosuccinimide (NBS). The batch experiment was done at constant temperature (70^{o}C) and time (90 min). The flask was placed in a thermostated water bath set at 30, 50, 70 and 90^{o}C, under atmospheric pressure according to operating conditions easily obtainable at commercial acetylation process. For each of the temperature range, the experiment was carried out at several reaction time (60, 90, 120, 150 and 180 min). Effect of catalyst was studied using several concentrations (0, 0.2, 0.4, 0.6, and 0.8 g) of N – bromosuccinimide (NBS) at constant sample dose, time and temperature. Keeping time, temperature and catalyst constant, effect of sample dose was studied by varying the dosages of the sample used. The flask was removed from the bath and the hot reagent was decanted off. The material was thoroughly washed with ethanol and acetone to remove unreacted acetic anhydride and acetic acid by – product. The new product was allowed to dry in an oven at 60^{o}C for 16 h, cooled and stored in a plastic container prior to analysis. The extent or level of modification of the sample material due to acetylation was estimated using three techniques; weight percent gain (WPG), extent of acetylation (R), and degree of substitution (DS).

Weight percent gain (WPG)

The WPG was determined by gravimetric technique using Adam analytical weighing balance (Model No. N17250) as described by Thompson *et al.* [11] and Azeh *et al.* [12]. It was calculated on the basis of oven – dried unreacted lignocellulosic fibers. The dried materials obtained were reweighed to determine the weight gains on the basis of initial oven dry measurements. Weight Percent Gain (WPG) of the materials due to acetylation was calculatedusing Equation 1:

$$\mathrm{WPG}(\%) = \left[\frac{\text{Weight Gain}}{\text{Original Weight}}\right] \times 100 \qquad (1)$$

Extent of acetylation (R)

The extent of acetylation was determined by the technique of Adebajo and Frost [2]. The extent of acetylation was estimated from Shimadzu-8400S model of Fourier Transform Infrared (FTIR) spectra by calculating the ratio (R) between the intensity of the acetyl C=O stretching band of esters at 1740 – 1745 cm^{-1} and the intensity of the C – O stretching vibrations of cellulose backbone at about 1020 – 1040 cm^{-1} (Equation 2). i.e.

$$R = \frac{I_{1740-1745}}{I_{1020-1040}} \qquad (2)$$

Degree of substitution (DS)

The percentage of acetylation (% acetyl) and degree of substitution (DS) was determined titrimetrically, following the method of Sodhi and Singh [13]. Acetylated sample (1.0 g) was placed in a 250 mL flask and 50 mL of 75 mL/100 mL 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 mol/L KOH was added. The excess alkali was back – titrated with 0.5 mol/L HCl using phenolphthalein as an indicator. A blank, using the original unmodified sample, was also used. Degree of substitution (DS) is defined as the average number of sites per glucose unit that possesses a substituent group.

$$\text{Acetyl}\,\% = \frac{[(\text{blank (mL)} - \text{sample (mL)}) \times \text{molarity of HCl} \times 0.043 \times 100]}{\text{weight of sample (g)}} \quad (3)$$

$$\text{DS} = \frac{(162 \times \text{acetyl (\%)})}{[4300 - (42 \times \text{Acetyl (\%)})]} \quad (4)$$

Statistical analysis

Multivariate (MANOVA) analysis explores outcomes from several parametric dependent variables across one or more independent variables [14]. The values of WPG, R and DS and how they respond to the variation of the different studied factors in this research were compared statistically with multivariate tests of SPSS version 16.

In this study, independent variables which are the factors (Sample dose, Catalyst amount, Time at 90°C, Time at 70°C, Time at 50°C, Time at 30°C) and dependent variables {weight percent gain (WPG), extent of acetylation (R) and degree of substitution (DS)} were compared using MANOVA**.** Pillai's Trace, Wilks' Lambda and Roy's Largest Root test statistics, were used in making decision on the MANOVA result since the independent variables are represented by more than two groups. The robustness of MANOVA result was tested using Box's M. It tests the variance – covariance matrices of the multiple dependent variables (WPG, R and DS) across the factors studied. Where the variance – covariance are equal, the robustness of MANOVA result is guaranteed [15]. Also, where there is statistical/significant difference in the multivariate analysis, Duncan's multiple comparison analysis was further used to determine the source of the difference.

Results and Discussion

Box's M test

The result in Table 1 indicates that the variance – covariance matrices of the techniques (WPG, R and DS) in all the materials, are non – significant or equal across the various factors considered. This is because Table 1 shows that the p –values for all the materials (0.061 for OPEFB, 0.063 for POBPs and 0.109 for CP) are greater than 0.001. This means that the vector of the techniques for all the materials, follows a multivariate normal distribution and therefore guarantee the robustness of any MANOVA result on the data.

Table 1: Box's test of equality of covariance matrices of techniques used in estimating acetylation across different factors for all the sample materials

	OPEFB	POBP	CP
Box's M	77.006	76.154	60.484
F	1.697	1.678	1.333
df1	30	30	30
df2	1.302E3	1.302E3	1.302E3
Sig. (p)	0.061	0.063	0.109

Multivariate analysis of variance (MANOVA)

Three test statistics (Pillai's Trace, Wilks' Lambda and Roy's Largest Root) were used in the decision of rejecting and accepting null hypothesis of MANOVA result. Table 2 showed that the p – values for all the test statistics used to analyze the response of the techniques to variation of the different factors during OPEFB, POBPs and CP acetylation, were greater than 0.05. Hence, null hypothesis was accepted which means that no significant/statistical difference in the response pattern of WPG, R, and DS towards variation of different factors considered in acetylating these materials. This suggests that WPG, R and DS are very sensitive to the determination of extent of acetylation, thus, indicating the reliability of gravimetric, Fourier Transform Infra–Red (FTIR) and titrimetric techniques for determining extent of acetylation as reported by Azeh *et al.* [12] Adebajo and Frost [2] and Sodhi and Singhi *et al.* [13], respectively. MANOVA results on the performance of techniques (WPG, R, and DS) at varied factors studied are also presented in Table 2. Comparing the techniques used in estimating extent of OPEFB and POBPs acetylation, three of the MANOVA test statistics are statistically significant ($p < 0.05$). This indicates that the null hypothesis is rejected, and thus, the difference between the performance of WPG, R and DS is statistically significant.

However, the three test statistics gave contradictory results when comparing the techniques used in estimating extent of CP acetylation. This could be as a result of difference in the statistical power of three tests statistics, with Roy's Largest Root having the most power when dependent variables (WPG, R, and DS) are highly correlated and the other two having the more power for more disparate outcomes [16]. Wilks' Lambda and Roy's Largest Root are often more powerful than Pillai's trace if the hypothesis degrees of freedom (h) are greater than unity ($h>1$) and one-dimension accounts for most of the separation among groups. Pillai's trace is more robust to departures from assumptions of MANOVA than the other three [15].

In this study, Pillai's Trace statistic lacks statistical significance while the other two are statistically significant. The hypothesis degree of freedom is greater 1 and no departures from assumptions and restrictions of MANOVA in our research data. Thus, we adopt Wilks' Lambda and Roy's Largest Root statistic results which indicate that significant difference exists between the performance of WPG, R and DS techniques. Our decision can also be supported by the fact that Wilks' Lambda and Roy's Largest Root statistics are used when the independent variables have more than two groups and for any number of groups respectively [17, 18]. Thus, in comparative evaluation of techniques used in estimating extent of lignocellulosic (OPEFB, POBP and CP) acetylation, all three test statistics provided evidence that the null hypothesis should be rejected, even though the values of the statistics and their degrees of freedom (h) vary (but greater than 1) because of their varying formulas and theoretical distributions [19].

Table 2: Multivariate tests results of techniques used in estimating acetylation for different effects and between techniques

Source		Value	F-test	Hypothesis df	Error df	Sig. (p)
Factors OPEFB	Pillai's Trace	1.378	.701	24.000	32.000	0.814
	Wilks' Lambda	.184	.485	24.000	18.653	0.952
	Roy's Largest Root	.594	.792[b]	6.000	8.000	0.601
Factors POBP	Pillai's Trace	1.376	.699	24.000	32.000	0.816
	Wilks' Lambda	.184	.486	24.000	18.653	0.952
	Roy's Largest Root	.661	.882[b]	6.000	8.000	0.548
Factors CP	Pillai's Trace	1.483	.786	24.000	32.000	0.727
	Wilks' Lambda	.144	.576	24.000	18.653	0.899
	Roy's Largest Root	1.317	1.756[b]	6.000	8.000	0.226
Techniques OPEFB	Pillai's Trace	1.869	18.991	12.000	16.000	<0.0001
	Wilks' Lambda	.000	2.387E2[a]	12.000	14.000	
	Roy's Largest Root	5.539E3	7.385E3[b]	6.000	8.000	
Techniques POBP	Pillai's Trace	1.656	6.414	12.000	16.000	<0.0001
	Wilks' Lambda	.000	4.068E2[a]	12.000	14.000	
	Roy's Largest Root	4.209E4	5.612E4[b]	6.000	8.000	
Techniques CP	Pillai's Trace	1.026	1.404	12.000	16.000	0.259
	Wilks' Lambda	.000	68.810[a]	12.000	14.000	<0.0001
	Roy's Largest Root	3.502E3	4.670E3[b]	6.000	8.000	<0.0001

Table 3: Tests of between-subjects effects and techniques

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F - test	Sig. (p)
Factors (OPEFB)	Sample Dose	799.777	4	199.944	.790	0.558
	Catalyst	194.515	4	48.629	.542	0.709
	Time at 90^{o}C	15.047	4	3.762	.220	0.921
	Time at 70^{o}C	93.510	4	23.377	.488	0.745
	Time at 50^{o}C	138.594	4	34.648	.435	0.781
	Time at 30^{o}C	8.190	4	2.048	.347	0.840
Factors (POBP)	Sample Dose	492.886	4	123.221	.110	0.976
	Catalyst	239.177	4	59.794	.064	0.991
	Time at 90^{o}C	62.635	4	15.659	.043	0.996
	Time at 70^{o}C	49.681	4	12.420	.111	0.976
	Time at 50^{o}C	33.891	4	8.473	.146	0.960
	Time at 30^{o}C	86.132	4	21.533	.189	0.939
Factors (CP)	Sample Dose	1210.581	4	302.645	.652	0.639
	Catalyst	143.663	4	35.916	.136	0.965
	Time at 90^{o}C	68.423	4	17.106	.202	0.931
	Time at 70^{o}C	49.771	4	12.443	.164	0.952
	Time at 50^{o}C	108.318	4	27.080	.241	0.908
	Time at 30^{o}C	87.193	4	21.798	.254	0.900
Techniques (OPEFB)	Sample Dose	935.792	2	467.896	2.344	0.138
	Catalyst	526.304	2	263.152	5.589	0.019
	Time at 90^{o}C	140.883	2	70.442	18.810	<0.0001
	Time at 70^{o}C	300.292	2	150.146	6.619	0.012
	Time at 50^{o}C	506.586	2	253.293	7.082	0.009
	Time at 30^{o}C	43.010	2	21.505	10.695	0.002
Techniques (POBP)	Sample Dose	10217.984	2	5108.992	42.659	<0.0001
	Catalyst	8797.995	2	4398.997	73.292	
	Time at 90^{o}C	3494.020	2	1747.010	115.658	
	Time at 70^{o}C	1026.213	2	513.106	41.799	
	Time at 50^{o}C	511.677	2	255.838	30.440	
	Time at 30^{o}C	960.265	2	480.133	21.550	
Techniques (CP)	Sample Dose	2209.812	2	1104.906	3.639	0.058
	Catalyst	2381.051	2	1190.526	34.681	<0.0001
	Time at 90^{o}C	711.344	2	355.672	20.986	<0.0001
	Time at 70^{o}C	650.995	2	325.498	24.861	<0.0001
	Time at 50^{o}C	889.536	2	444.768	15.652	<0.0001
	Time at 30^{o}C	691.150	2	345.575	16.387	<0.0001

Table 3 also confirmed the decision of the three test statistics, on the performance of the techniques and how they responded to the variation of all the factors considered in acetylating the sample materials. However, the p – values of sample dose factors for OPEFB and CP acetylation showed that there is no significant difference in the performance of the techniques but the test statistics suggests otherwise which is accepted.

Post hoc (Duncan multiple range test) analysis

MANOVA has post hoc procedures to determine why the null hypothesis was rejected.

Thus, when a significant difference is found using analysis of variance, we determine the source of the difference. It is therefore necessary to conduct post hoc comparisons.

The MANOVA results which suggest existence of significant/statistical difference between the techniques, were further analyzed using Duncan's multiple range tests analysis. This is to determine the best technique used in estimating extent of acetylation and which significantly performed better than others. Tables 4, 5 and 6 present Duncan multiple range tests results on the performance of the techniques (WPG, R and DS) used in estimating extent of acetylating the selected lignocellulosic materials. In all the factors considered as shown in the Tables, WPG is classified in a subset different from the other two techniques.

Table 4: Multiple comparison (Duncan) analysis of ANOVA for OPEFB where P = α < 0.05

Techniques	N	Subset for alpha = 0.05	
		1	2
Effect of Catalyst			
DS	5	.4080	
R	5	.8740	
WPG	5		**13.2000**
Sig.(p)		.916	**1.000**
Effect of Time at 90°C			
DS	5	.5300	
R	5	.8820	
WPG	5		**7.2000**
Sig. (p)		.779	**1.000**
Effect of Time at 70°C			
DS	5	.2620	
R	5	.7760	
WPG	5		**10.0000**
Sig. (p)		.867	**1.000**
Effect of Time at 50°C			
DS	5	.1720	
R	5	.7960	
WPG	5		**12.8000**
Sig. (p)		.872	**1.000**
Effect of Time at 30°C			
DS	5	.0840	
R	5	.8580	
WPG	5		**4.0000**
Sig. (p)		.405	**1.000**

Table 5: Multiple comparison (Duncan) analysis of ANOVA for POBP where P = α < 0.05

Techniques	N	Subset for alpha = 0.05	
		1	2
Effect of Sample Dosage			
R	5	.8140	
DS	5	.8540	
WPG	5		**56.2000**
Sig. (p)		.995	**1.000**
Effect of Catalyst			
DS	5	.5740	
R	5	.6760	
WPG	5		**52.0000**
Sig. (p)		.984	**1.000**
Effect of Time at 90°C			
DS	5	.8080	
R	5	.8400	
WPG	5		**33.2000**
Sig. (p)		.990	**1.000**
Effect of Time at 70°C			
DS	5	.8300	
R	5	.8780	
WPG	5		**18.4000**
Sig. (p)		.983	**1.000**
Effect of Time at 50°C			
DS	5	.5520	
R	5	1.0860	
WPG	5		**13.2000**
Sig. (p)		.776	**1.000**
Effect of Time at 30°C			
DS	5	.2720	
R	5	1.0060	
WPG	5		**17.6000**
Sig. (p)		.810	**1.000**

Table 6: Multiple comparison (Duncan) analysis for MANOVA result of CP where P = α <0.05

Techniques	N	Subset for alpha = 0.05	
		1	2
Effect of biosorbent dosage			
DS	5	.7440	
R	5	.9620	
WPG	5		**26.6000**
Sig. (p)		.985	**1.000**
Effect of catalyst			
DS	5	.7640	
R	5	.9840	
WPG	5		**27.6000**
Sig. (p)		.954	**1.000**
Effect of Time at 90°C			
DS	5	.9360	
R	5	1.0480	
WPG	5		**15.6000**
Sig. (p)		.966	**1.000**
Effect of Time at 70°C			
DS	5	.5960	
R	5	1.0660	
WPG	5		**14.8000**
Sig. (p)		.841	**1.000**
Effect of Time at 50°C			
DS	5	.6900	
R	5	1.0440	
WPG	5		**17.2000**
Sig. (p)		.918	**1.000**
Effect of Time at 30°C			
DS	5	.5400	
R	5	1.0760	
WPG	5		**15.2000**
Sig. (p)		.857	**1.000**

Thus, the Tables (i.e., Tables 4, 5 and 6) showed that WPG is the source of the significant difference when comparing the performance of the three techniques used in estimating extent of acetylation in the selected materials. There is no significant difference between R and DS, however there is significant difference between WPG and the other two techniques (R and DS). The p-values of the subset containing the WPG for all the factors considered, is higher than that of the subset containing the other two techniques. This indicates that WPG performed better than the other two techniques in estimating extent of acetylation under several factors considered.

Conclusion

Homogeneity tests showed that MANOVA results are very robust since the sample sizes for the materials are equal. Multivariate analyses suggest that any of the techniques (WPG, R and DS) can be used to estimate extent of acetylation. It indicated that the techniques do not differ significantly in their response to the variation of several factors considered in acetylating lignocellulosic materials. However, the performance of these techniques in estimating extent of acetylation, differs significantly. Duncan's post hoc analyses suggested that WPG performed differently and better than other two techniques in estimating extent of acetylation. Thus, extent of acetylation is better estimated using gravimetric technique which is cost effective and easy to carry out.

Conflict of interest: The authors declare that they have no competing interests. All authors have endorsed the publication of this research.

Acknowledgments: We are grateful to the entire Staff of Chemistry Department, Ahmadu Bello University, Zaria; for their support during the course of this research work. Special thanks also go to Mr. Ochigbo and his team for their technical support. Mahmud Aboki of National Research Institute for Chemical Technology (NARICT) Zaria, Kaduna – Nigeria is acknowledged for his assistance in the running of FTIR machine.

References

[1] Bello–Pérez, L. A., Agama–Acevedo, E., Zamudio–Flores, P. B., Mendez–Montealvo, G. and Rodriguez–Ambriz, S. L. (2010). Effect of low and high acetylation degree in the morphological, physicochemical and structural characteristics of barley starch. *LWT – Food Science and Technology*, 43, 1434–1440. https://doi.org/10.1016/j.lwt.2010.04.003

[2] Adebajo, M. O. and Frost, R. L. (2004). Acetylation of raw cotton for oil spill cleanup application: an FTIR and 13C MAS NMR spectroscopic investigation. *Spectrochim. A. Part A: Mol. Biomol. Spectrosc*., 60(10), 2315-2321. https://doi.org/10.1016/j.saa.2003.12.005

[3] Stevens, J. P. (2002). Applied multivariate statistics for the social sciences. Mahwah, NJ: Lawrence Erlbaum Associates.

[4] Zientek, L. R. and Thompson, B. (2009). Matrix summaries improve research reports: Secondary analyses using published literature. *ER*., 8, 343–352. https://doi.org/10.3102/0013189X09339056

[5] Thompson, B. (2006). Foundations of behavioral statistics. The Guilford Press, New York, NY.

[6] Huberty, C. J. and Morris, J. D. (1989). Multivariate analysis versus multiple univariate analyses. *Psychol. Bull*., 105, 302 – 308. https://doi.org/10.1037/0033-2909.105.2.302

[7] Thomas, D. R. (1992). Interpreting discriminant functions: A data analytic approach. *Multivariate Behav. Res*., 27, 335 – 362. https://doi.org/10.1207/s15327906mbr2703_3

[8] Whitely, E. and Ball, J. (2002). Statistics review 5: Comparison of means. *Crit. Care*. 6, 424 – 428. https://doi.org/10.1186/cc1548

[9] Bewick, V., Cheek, L. and Ball, J. (2004). Statistics review 9: One-way analysis of variance. *Crit. Care*., 8(2), 130 – 136. https://doi.org/10.1186/cc2836

[10] Sun, X. F., Sun, R. C. and Sun, J. X. (2004). Acetylation of sugarcane bagasse using NBS as a catalyst under mild reaction conditions for the production of oil sorption – Active materials. *Bioresour. Technol*., 95(3), 343 – 350. https://doi.org/10.1016/j.biortech.2004.02.025

[11] Thompson, N. E., Emmanuel, G. C., Adagadzu, K. J. and Yusuf, N. B. (2010). Sorption studies of crude oil on acetylated rice husks. *Arch. Appl. Sci. Res*., 2(5), 142 – 151.

[12] Azeh, Y., Olatunji, G. A., Mohammed, C. andMamza P. A. (2013). Acetylation of wood flour from four wood species grown in Nigeria using vinegar and acetic anhydride. *Int. J. Carbohydr*., 3, 456 – 467. https://doi.org/10.1155/2013/141034

[13] Sodhi, N. S. and Singh, N. (2005). Characteristics of acetylated starches prepared using starches separated from different rice cultivars. *J. Food Eng.*, 70, 117 – 127. https://doi.org/10.1016/j.jfoodeng.2004.09.018

[14] Hinkle, D. E., Wlersma, W. and Jurs, S. G. (1998). Applied Statistics for the Behavioral Sciences. Houghton, Mifflin, and Boston; https://doi.org/10.3102/10769986015001084

[15] Tabachnick, B. G. and Fidell, L. S. (2001). Using Multivariate Statistics. 2nd ed. Harper Collins, New York.

[16] Huberty, C. J. and Olejnik, S. (2006). Applied MANOVA and discriminant analysis. John Wiley & Sons. Hoboken, New Jersey. https://onlinelibrary.wiley.com/doi/book/10.1002/0471178947X

[17] Brace, P., Kevin, A., Martin, J. and Stacy, G. U. (2006). Reprint of C: Does state political ideology change over time? *Polit. Res. Q.*, 59, 493–4.

[18] Coakes, S. J. and Steed, L. G. (2007). SPSS: Analysis without anguish: Version 14.0 for Windows. Brisbane: John Wiley & Sons Australia Ltd. https://archive.org/details/spssversion140fo0000coak

[19] Haase, R. F. and Ellis, M. V. (1987). Multivariate analysis of variance. *J. Couns. Psychol*., 34, 404 – 413. https://doi.org/10.1037/0022-0167.34.4.404

Citing this Article

Onwuka, J. C., Akeyede, I. & Jasper, E. E. (2023). Comparative evaluation of analytical techniques used in estimating acetylation of lignocellulosic materials. *Lafia Journal of Scientific and Industrial Research*, *1*(1&2), 12 – 17. https://doi.org/10.62050/ljsir2023.v1n1.265