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STUDIES IN NITRATION OF JUTE, FLAX, COTTON AND OTHER VEGETABLE FIBRES UNDER DIFFERENT TREATMENTS

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Studies in nitration of jute, flax, cotton and other vegetable fibres were conducted at Lyallpur during the two years 1965-66 and 1966-67 and values were found to range from 110.880 to 140.270%. The highest value was recorded by sunhemp, followed by calotropis, $jute_1$, $jute_2$, $kenaf_2$, $kenaf_1$, bombax cotton, patwa, AC134 and L.S.S., where the lowest value was observed in case of flax followed by ramie, AC307 and 4F. The varietal differences whereas found to be highly significant at all the stages of maturity. The spacing effects were significant at flowering and seed maturity stages and non-significant at pre-flowering stage. The manurial effects were highly significant at flowering, significant at seed maturity and non-significant at pre-flowering stage. A general decrease in the values of nitration percentage was observed in all vegetable fibres from the pre-flowering to the seed maturity stage and therefore, superior quality fibres for industrial purposes can only be obtained at the pre-flowering stage.

Introduction

The industrial value of cellulose nitrates first prepared by Schonbein (Worden 1921, 1942), Fabel 1941) is already well known. These are prepared by action of cencentrated nitric acid on cellulose which penetrates into the chains of cellulose and directly acts on hydroxyl groups. This reaction usually reaches equilibriun very soon and the degree of nitration is low. The percentage of nitrogen in the product usually ranges from 6.5% with 28.4% water to 13.65% water in the reaction mixture and therefore, the degree of nitration can be controlled by adjustments in the water content of the nitrating mixture. The introduction of concentrated sulphuric acid normally enhances this action as it acts as a dehydrating and swelling agent. The equiliberium may thus be shifted towards right and a cellulose nitrate of a high degree of nitration is obtained.

The degree of nitration is further known to affect solubility of the nitrate and it is possible to prepare a nitrate of any designed solubility by controlling the degree of esterification through adjustments in the amount of water in the reaction mixture. The group of nitrates with nitrogen content higher than 12.8% which are practically insoluble in water are known as gun cotton, whereas nitrates completely soluble in mixed solvent and with nitrogen content of 11–12.5% are termed as 'Collodian'. On the other hand cellulose nitrates with 10–11% nitrogen are very suitable for nitrates compounding with camphor or other plasticizers for use as plastic.

In view of the great industrial value of cellulose nitrates, it was considered desirable to conduct regular studies in nitration of jute, flax, cotton and some other available vegetable fibres at Lyallpur. Very interesting results were obtained, which are presented in this paper.

Review of Literature

Some research work pertaining to the nitration of vegetable fibres has been reported by a few workers from some countries and a brief review of the relevant literature is given here:

Worden (1921, 1942) and Fabel (1941) stated that Schonbein was the first to prepare cellulose nitrates which have great industrial value in the present day world.

Whitford[1] reported that the nitration value of calotropis gigantea was 153.0%.

Goldthwait[2] and Guthrie[3] stated that the nitration percentages of jute, sunhemp, ramie and flax were 128.0, 150.5, 125.0 and 123.0% respectively and these findings were supported by Matthews and Mauersberger.[4]

Material and Methods

The present studies were carried out at the West Pakistan Agricultural University, Lyallpur, during the two years 1965-66 and 1966-67. The experiment was laid out in a split plot design with three replications, six varieties of bast fibres, three spacings and two manurial treatments. The details of the experiment are given here:

Varieties.—Jute=J_1 (*Corchorus oltorius* L.); Jute=J_2 (*Corchorus capsularis* L.); Kenaf=K_1 (*Hibiscus cannabinus* L. Var Viridis); Kenaf=K_2 (*Hibiscus cannabirus* L. Var Vulgaris); Patwa=P (*Hibiscus subdariffa* L.); Sunhemp=S.H (*Crotalaria juncea* L.).

Spacings.—

Row to row distance	Plant to plant distance
S_1= 9 in	4—6 in
S_2=12 in	4—6 in
S_3=15 in	4—6 in

Fertilizer Rate Per Acre.—M^0=Control; M_1= 50 lb of nitrogen/acre.

Pure samples of cotton 4F, L.S.S., AC134 and AC307 from control and 50 lb of nitrogen/acre were also collected. The samples of flax, ramie, calotropis and bombax cotton were collected from different sources for the present studies.

Pure seed of all the varieties was taken from the Department of Plant Breeding and Genetics, West Pakistan Agricultural University, Lyallpur. The seed was sown on the 15th of April during both the years, according to the well designed layout plan. The experimental crop received normal and uniform agricultural operations during the growing period. Nitrogen was applied as ammonium sulphate 1½ month after sowing when the seedlings were 9–12 in high. The condition of crop during both the years was normal.

Sampling.—five lb samples from each plot was taken at the following three stages of maturity: (1) Preflowering; (2) Flowering; (3) Seed maturity.

Retting.—The samples thus taken were subjected to water retting under closed tank system for the separation of the fibres from the woody core of the stem. The fibres were separated by mannual labour, dried under shade and were studied in the laboratory to see the effect of fertilizer, spacing and varieties on quality of fibres.

Methods Used.—The standard methods for the collection of the most authenticated observations relating to nitration were the same as suggested by Matthews *et al.*[5] which entail the full details of the formation of nitrocelluloses.

Nitration is represented by the increase in wt sustained by the fibres when treated for over 1 hr with a mixture of equal volumes of both nitric acid and sulphuric acid.

A composite sample free from impurities is prepared and finely divided. 2-3 g of sample is treated with 10 cc of the acid mixture at 40°C for 1½ to 2 hr. It is then washed with distilled water to remove the excess acid, filtered by giving subsequent washings and dried to 30% moisture. Finally it is weighed and nitration percentage is calculated on the basis of oven dry wt of the sample.

Statistical Analysis.—The data thus collected were analysed statistically by the analysis of variance method described by Snedecor.[5] The treatment means were compared using L.S.D. test method of significance (Leclerg *et al.*).[6]

Results and Discussion

The results of present studies relating to nitration of jute, flax, cotton and some other vegetable fibres, conducted at Lyallpur, during the years 1965–66 and 1966–67, are presented in Tables 1–4 and are discussed here.

It will be observed from the data presented in Tables 1–2 that the verietal effects were found to be highly significant at all the stages during both the years. It will be clear from varietal means that jute,1 was at par with Jute2 and kenaf1 was at par with kenaf2 at pre-flowering and flowering stages, while at seed maturity stage these were found to differ significantly from each other and highly significantly from Roselle (patwa) and sunhemp.

The sumhemp recorded the highest values of 140.03 and 140.270% at pre-flowering stage during the 1st and 2nd year respectively, whereas the lowest value of 128.189 and 126.34% at the seed maturity stages were recorded in the case of

TABLE 1.—SHOWING ANALYSIS OF VARIANCE OF NITRATION.

Variation due to	F. Ratios					
	1965-66			1966-67		
	Pre-flowering	Flowering	Seed maturity	Pre-flowering	Flowering	Seed maturity
Manures	5.865 NS	1388.462**	23.656*	8.322 NS	104.657**	44.751*
Spacings	2.855 NS	7.970*	8.303*	2.764 NS	5.837*	6.989*
Varieties	268.442**	293.261**	359.822**	610.711**	827.299**	535.644**

** Highly significant; * Significant; N.S.=Non-significant.

TABLE 2.—SHOWING STATISTICAL SUMMARY OF THE MAIN TREATMENTS.

Treatment	Means 1965–66			1966–67		
	Pre-flowering	Flowering	Seed maturity	Pre-flowering	Flowering	Seed maturity
Varieties						
K_1	137.200	134.378	130.733	137.43	134.336	130.470
K_2	137.220	134.406	130.778	237.45	134.342	130.520
J_1	138.130	135.167	131.266	138.09	135.116	131.030
J_2	138.100	135.139	131.240	138.060	135.096	130.870
P_1	133.34	130.178	128.189	133.06	130.044	126.34
S.H.	140.03	136.539	132.594	140.270	136.511	132.23
Spacings						
S_1	137.348	134.383	130.847	137.399	134.387	130.303
S_2	137.341	134.319	130.789	137.395	134.353	130.241
S_3	137.331	134.244	130.722	137.390	134.310	130.191
Manures						
M_0	137.24	134.57	130.726	137.37	134.010	130.190
M_1	137.44	134.473	130.846	137.42	134.490	130.300
C.D. Spacings						
5%	0.0187	0.0379	0.0977	0.0504	0.0410	0.0879
1%	0.0363	0.0735	0.1894	0.0978	0.0796	0.0700
C.D. Varieties						
5%	0.178	0.091	0.190	0.084	0.034	0.014
1%	0.251	0.128	0.268	0.118	0.048	0.057

TABLE 3.—SHOWING ANALYSIS OF VARIANCE OF NITRATION.

Variation due to	D.F.	S.S.	M.S.	F. ratio	t. value
Samples	3	205.4931	68.4977		
Varieties	3	1567.1546	522.3849	5.44	*
Error	9	932.0883	103.5653		
Total	15	2704.7360			

* = Significant

Statistical summary of the main treatments

Varieties

S.E. = 5.088
S.E.D.M. = 7.1955
Cd1 =27.775
Cd_2 =50.297

Calotropis	*Bombax Cotton*	*Ramie*	*Flax*
138.818	134.615	120.123	110.880

TABLE 4.—SHOWING ANALYSIS OF VARIANCE OF NITRATION.

Variation due to	D.F.	S.S.	M.S.	F. Ratio	t. Value
Samples	3	8.932385	2.977462		
Varieties	3	19.999610	6.666537	2.144	NS
Manures	1	0.000003	0.000003	0.0000009	NS
V x M	3	0.030734	0.0102447		
Error	21	65.300890	3.109566		
Total	31	94.263622			

NS. = Non-significant

Varieties

AC134	L.S.S.	4F	AC307
133.009	133.008	132.144	131.086

Manures

Mo	*M*1
132.311	132.312

patwa during the 1st and 2nd year respectively. The actual values for sunhemp, $Jute_1$, $Jute_2$ $kenaf_2$ $kenaf_1$ and patwa at pre-flowering stage were 140.03, 138.130, 133.100, 137.220, 137.200 and 133.34% respectively and similar trends were recorded during both the years. There was a progressive decline with the progress of maturity.

It will be clear from Table 3 that the varietal differences were significant and the highest value of 138.88% was recorded by calotropis followed by 134.615, 120.123 and 110.880% in the case of bombax, ramie and flax respectively. Both varietal and manurial effects on value of nitration of cotton varieties were found to be non-significant, however, the highest value of 133.009% was recorded by AC134 followed by 133.008, 132.144 and 131.086% from L.S.S., 4F and AC307 respectively.

It will be clear from Tables 1 and 2 that the spacing effects were non-significant at pre-flowering stage but the effects were significant at flowering and seed maturity stages. At flowering and seed maturity stages during 1965–66, S_1 and S_2 were at par and S^1 was significantly different from S_3 which was in turn at par with S_2, whereas during the year 1966–67, S_3 was significantly different from S_1, and S_2 at flowering stage, which were at par with each other. By the application of nitrogenous fertilizers to the crops, the nitration value increased but with increase in the row to row distance, it decreased slightly.

It will be seen from Tables 1 and 2 that the nitrogenous fertilizers affected nitration non-significantly at the pre-flowering stage, highly significantly at flowering and significantly at seed maturity stage during both the years.

Guthrie,[3] Goldthwait[2] and Matthews and Mauersberger[4] reported the nitration percentage of jute, sunhemp, ramie and flax to be 128.0, 150.5, 125.0 and 123.0 respectively, whereas the present studies have clearly indicated that the values for nitration ranged from 131.020 to 138.13% for jute, and 130.870 to 138.00% in the case of $jute_2$ and in the case of sunhemp the value was 132.23–140.270% and for ramie and flax the values were found to be 120.123 and 114.880% respectively, which show a higher trend in the case of sunhemp, whereas all the other types were found to possess lower values than those reported by the earlier workers. Whitford[1] gave nitration value of 153.0% for calotropis whereas, it was 138.818 in the present studies.

The increase in wt with treatment of fibre material for 1½ to 2 hr with a mixture of equal volumes of nitric and sulphuric acid, results in a cellulose nitrate of a high degree of nitration, which affects the solubility of the nitrates and it has been put to great industrial uses in the manufacture of gun cotton, colloidian and plastics.

The present investigations have shown that the range of nitration percentage was found to be 110.880 to 140.270 in all vegetable fibres with the highest range of 132.23 to 140.270 in sunhemp; 138.818 for calotropis; 130.870 to 138.130 in jute types; 130.470 to 137.450 in kenaf varieties; 134.615% in bombax; 126.34–133.34% in patwa;

131.286 to 133.009% in cotton varieties; 120.123% in ramie and 110.880% in flax.

The range of nitration in kenaf, jute, patwa and sunhemp was the highest at pre-flowering stage 133–06.140.270%); 130.0441:–6.5329% at flowering stage and 126.34 to 132.594 at the seed maturity stage. The harvesting of vegetable fibres for industrial purposes may be done at the pre-flowering stage only. Close spacing recorded higher and significant values of nitrate at flowering and seed maturity stages whereas the manurial effects although significant at flowering and seed maturity stages.

Summary

Studies in nitration of jute, flax, cotton and other vegetable fibres were conducted at Lyallpur, during the years 1965-66 and 1966-67.

The varietal differences were found to be highly significant at all the stages of maturity. The values ranged from 114.880 to 140.270% the lowest value was given by flax and the highest value was obtained in case of sunhemp, followed by calotropis, $jute_1$ and $jute_2$ with actual values of 138.818, 138.130 and 138.100% respectively.

The spacing effects were significant at flowering and seed maturity stages and were non-significant at pre-flowering stage, whereas the manurial effects were highly significant at flowering, significant at seed maturity and non-significant at pre-flowering stages.

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