Organosolv Pulping of Agave Sisalana

*Kulkarni H G , *Sirmokadam N.N., *Shenvi A.K., *Hegde G.M., *Padwalkar S.N, & *Guha S.R.D.

ABSTRACT

The paper records the results of an investigation on the pulping of Agave sisalana by organosolv pulping process under atmospheric conditions using 1:1 aqueous ethanol and 2% alkali at 80° C. for 4 hours cooking. The unbleached pulp yield was found to be 88.6% with Kappa Number of 37.0 The strength properties of the unbleached pulp were found satisfactory and comparable with Agave sulfate pulp. It is a long fibred and promising alternative source for pulp and paper industry. A high recovery of solvent and low pollution are the distinctive features of this process.

Introduction :

Conservation of forests, utilization of waste lands and providing alternate and suitable fibrous raw material to paper industry are some of the issues on the national agenda. Efforts are being made to develop technologies to manufacture paper utilizing available non-wood fibrous raw materials like agricultural and horticultural residues/wastes.

Sisal, a non-wood xerophytic plant of Amaryllidaceae family which exists in various species. The genus Agave consists of about 250 species. There are 5 species of Agave prominently grown in India and are :

(i) Agave sisalana, (ii) Agave wightii, (iii) Agave cantala (Roxb), (iv) Agave Americana (Linn), and (v) Agave Vera–Cruz. Agave sisalana commonly 'known as 'Sisal' is one of the promising and good sources of fibrous raw materials.

The sisal plants are grown in areas with high temperature and an annual rain fall of 1200 to 1800 mm. It is planted on barren hill slopes to check soil erosion. The sisal plant has an eight-year. growth cycle, at the end of that time the mother plant flowers and dies. The Agave growth is propagated either by rhizomes or bullbills developed in the nursery over a period of about one year. During its productive period it gives from 180 to 240 leaves. Sisal yields upto 2800 kg fibre per hectare under optimum conditions.

Sisal is a native of Mexico (Yucatan) and has successfully thrived in the semi-arid regions of the tropics. In Brazil, it has been cultivated for over 30 years. Brazil is the largest producer of sisal followed by Tanzania and kenya. plantings of sisal hemp have been started in Brazil to supply raw material for an annual production of 64,000 tons of high-quality, bleached long-fiber pulp.

It has become possible to separate the biomass into cellulosic fibres suitable for pulp and papermaking by the use of chemical delignifying processes such as kraft, soda, sulfite and recently organosolv. A chemical technology which could achieve very less pollution, easy chemical recovery and selective delignification and could provide an integrated approach for the efficient utilization of the biomass is at our disposal and that is organosolv pulping process which is based on organophillic nature of lignin. Due to low surface tension. organic solvents can penetrate faster into the cell wall and thus achieve efficient hydrolysis of lignin-carbohydrate bonds. This insight in reaction mechanism formed the genesis of organosolv technology in the production of pulp and paper.

The following aims form the background to an increasing diversification of the pulping process :-

— improved pulp yields,

— reduction of energy consumption.

*Bangur Nagar Degree College, Dandeli —581 325

**Central Laboratory, W.C.P.M. Ltd., Dandeli—581 325

— reduction of the amounts of chemicals required for pulping and bleaching, including improved recovery process for the chemicals.

— reduction of air and water pollution.

— development of sulfur-free pulping processes ond chlorine free bleaching sequences.

— high flexibility with regard to pulp yield, quality and bleachability.

— process conditions allowing the suitable preparation of pulping by-products.

— profitable smaller production units requiring lower costs for new mills and reduced raw material demand.

An Overview of organosolv pulping :

A number of organosolv delignification methods have been explored by a large number of investigators using various organic solvents for avoiding loss of carbohydrate during delignification. Among the known organic solvents, alcohols and amines have generated increasing interest.

The use of ethanol in organosolv delignification was first studied and developed by kleinert and Tayentha 1[1]. Arnovsky and Gortner indicated n-butanol as an effective solvent for delignification in place of ethanol[2]. Lately Diabold et al[3] claimed a US Patent based on plurality of extraction of lignin from the wood using ethanol,

The pioneering work of ethanol delignification in the presence of acid catalyst was carried out by Sarkanen et.al[4]. In order to avoid the inherent pollution problem associated with sulfides, aqueous ethanol delignification was first attempted in the presence of alkali and in the absence of sulfide independently by Marton et.al[5] and April et al[6].

The growth of amine solvent extraction took place very much in parallel to the advance of alcohol solvent extraction. Ethanolamine and ethylene diamine were found to be good delignifying agents as early as 1940. Peterson and wise patented the process[7] The use of ethylene diamine in combination with alkali pulping was attempted by two groups, Julien et al in the United States[8] and kubes et.al in Canada[9], Ketzen and co-wrokers[10] showed that pulping with ethanol is an economically viable alternative to the conventional processes. They have profounded Alcohol pulping and Recovery process (APR Process) for a hypothetical commercial plants. The plant uses stationary extractors and alcohol recovery system. In early eighties, a couple of agencies have developed process for organosolv pulping. A few of them are ALCELL process (formerly known as APR Process) developed by Repal Technologies, Inc BEC process, developed by Biological energy corporation, valley Forge, pennsylvania and the MD process developed by MD company Munich, west Germany.

This prompted the authors to explore the possiblity of using Ethanol-Ethylane diamine and Aqueous Ethanol-Alkali (NaOH) for selective delignification of Indian raw materials. As an initial step, rice straw[11] and Arecanut husk[12] were taken up for investigation by organosolv pulping.

Experimental :

Agave leaves usually 0.75 to 1.5 metre in length and 0.1 to 0.15 metre in width, weighing about 0 5 to 0.7 kg and containing 80–85%moisture, 4.5% fibre content and 2.5% pith were procured in green condition from nearby Kherwada village. The sisal fibres were isolated from the leaves by the prehydrolysis method given below :

The fresh agave leaves were crushed and the analysis of juice (mucilage) collected is in progress for isolation of Hecogenin which is valuable for the partial synthesis of the drug 'cortisone' The leaves after crushing were cut into chips of average size 6 cm × 6 cm. The chips were heated in a digester with sufficient water at 100°C. for 2 hours. The softened chips were refined through sprout waldron Disc Refiner. Adhering pith from fibres was removed by washing it over a flat screen.

Proximate Analysis

The proximate analysis of the sisal fibre was done as per TAPPI Standard Testing Methods. Even the fibre dimensions were also determined. The results are recorded in Table I.

Pulping of Sisal Species

The pulping experiments were carried out to optimise the conditions. Each batch consisted of 50 gms material which was subjected to organosolv

pulping using 1 : 1 aqueous ethanol and at 80°C. The variable parameters like bath ratio, alkali charge and time of cooking were studied and the results are indicated in Table II, III and IV.

TABLE–I
Proximate Analysis

Sl. No	Particulars		
1	Cold water solubility	%	32.8
2	Hot water solubility	%	36.1
3	1% NaOH solubility	%	51.8
4	Ash	%	7.2
5	Alcohol-benzene solubility	%	12 0
6	Holocellulose	%	69.0
7	Lignin	%	11.4
8	Pentosans	%	11.2
9	Average Fibre length	mm	3.2
10	Average Fibre diameter	microns	16 0
11	Slenderness ratio	Length/ diameter	200

TABLE–II
Effect of Bath Ratio on Pulping

Cooking conditions : (a) NaOH : 2%
(b) Cooking temperature : 80°C.
(c) Cooking time : 4 Hours

Sl.No.	Bath Ratio	Kappa No.	Yield, %
1	1 : 3)	Results are unsatisfactory due to	
2	1 : 4)	partial impregnation	
3	1 : 5	40	91
4	1 : 6	37	88
5	1 : 7	35	84
6	1 : 8	33	80
7	1 : 10	30	76

TABLE–III
Effect of Alkali Charge on Pulping

Cooking conditions :
(a) Bath ratio : 1:6
(b) Cooking temperature : 80° C.
(c) Cooking time : 4 Hours

Sl.No.	Alkali Charge %	Kappa Number	Yield %
1	2	37	88
2	4	34	84
3	6	32	79
4	8	29	73

TABLE–IV
Effect of Time on Pulping

Cooking conditions :
(a) Bath ratio : 1:6
(b) NaoH : 2%
(c) Cooking temperature : 80°C

Sl.No.	Cooking Time Hours	Kappa Number	Yield %
1	1	41.0	93
2	2	40 0	93
3	3	38 5	92
4	4	37.0	88
5	5	35 0	83
6	6	33.0	78

From the studies of variable parameters, we arrived at optimum conditions of pulping i.e. 1.6 bath ratio 2% alkali charges and 4 hours cooking at 80°C. The 300 gm material cooked in the 5 litres digester using above conditions was subjected to mechanical action by running in the Valley beater to get uniform pulp of freeness 40°SR. The cooking conditions of pulping and the strength properties of unbleached organosolv pulp indicated in Table V was compared with conventional sulfate pulp[13]. The organosolv lignin from spent black liquor after recovery of the solvent to the extent of 92%, was precipitated by adjusting the pH to 4.3 and its study of utilisation is in progress.

TABLE–V
Cooking Conditions and Results of Unbleached Organosolv Sisal Pulp Compared with Sulphate Pulp

Sl. No.	Particulars		Organosolv cook	Sulphate cook
1	Alkali as NaOH	%	2.0	—
2	Alkali as Na_2O	%	—	10.0
3	Bath ratio		1:6	1:5
4	Cooking time	Hrs.	4.0	1 0
5	Cooking temperature	°C.	80.0	165.0
6	Yield	%	88.6	66.6
7	Residual Alkali (as NaOH)	Gpl	—	—
8	Residual Alkali (as Na_2O)	Gpl	—	7.0
9	Kappa Number		37.0	25.5
10	Viscosity (CED)	CPs.	32.4	—
11	Strength Properties at 40° SR :			
a)	Burst Factor		29.7	42.5
b)	Breaking Length	Mtrs.	5267	7040
c)	Tear factor		188 0	130.0
d)	Double Folds (MIT)	Nos	509 0	583.0

Results and Discussion

The proximate analysis indicated that the lignin content (11 4%) is less and holocellulose content (69 0%) is more compared to bamboo and hardwoods. Tne fibre morphological studies revealed that it is long fibered material and so can be used for making strong paper.

From Table No. IIit was noticed that higher the Bath Ratio lower will be the yield and Kappa No. Furtner, it was concluded that 1:6 bath ratio gave satisfactory delignification with a Kappa No of 37 and with an yield of 88%.

The presence of alkali provides needed basicity to bring out the required delignification. However higher concentration of OH ions leads to peeling reaction and hampers the yield. Table III indicates the variation in the alkali charge. Higher the alkali charge, lesser will be Kappa No. and yield. It wns concluded that the addition of just 2% alkali on O D. basis gave satisfactory results with kappa No.37 and with 88% yield.

The cooking time was from 1 to 6 hours keeping the Bath ration 1 : 6 and 2% alkali charge. It is evident from the results of Table IV that lower kappa No. and yield is achieved with tha increasing of the cooking time of 4 hours.

The burst factor and breaking length of unbleached organo–solv pulp are comparable to Agave karft pulp while the tear factor is comparable to even softwood pulps. This can be attributed to longer fibre length (3.2 mm).

Conclusion :

Since the alkali charge is less and the recovery of solvent is good and the yield, and strength properties of the sisal fibre are comparable to conventional sulfate process, therefcre organosolv pulping of sisal would be economically viable and environmentally acceptable. However, the overall economics depends upon the individual mill's requirements, proximity to the availability of the material and the facilities available to handle this bulky material ete.

Owing to its high tearing strength and other features like porosity, high bnlk absorbency and folding endurance, the sisal pulp can also be mixed with other pulps to reinforce certain strength properties to papers produced.

Acknowledgement :

The authors are grateful to Karnataka State Council for Scienc and Technology (KSCST), Bangalore for financial assistance. They are thankful to shri J. S. Sanwal, General Manager (Opn), W.C.P.M. Ltd. for his valuable suggestions. Further, their sincere thanks go to Managements of W.C.P.M. Ltd., Dandeli and of Dandeli Education Society, Dandeli and to the Principal and staff of Bangur Nagar Degree College, Dandeli (N.K.). They are also thankful to shri A.S. Bhat, forest Manager, W.C.P.M. Ltd. for giving valuable information on Agave.

References :

1 Kleinert, T.N., Tayenthal K.Z. Angew Chem-, 44:788 (1931)

2 Arnovosky, S.I , Gortner, R.A , Ind. Eng· Chem., 28:1270 (1936)

3 Diebold, V.B., Cowan, W.F., Walsh, J.K. U S. Patent 4,100,016, July 11, 1978.

4 Sarkanen, K V , Assiz, S., Chang, V, Paper Presented at the 98th AICHE Meeting, Portland, Oregon (1980).

5 Morten, R. Granzow, S Tappi 65 (6) : 103—106 (1982).

6 April, G.C. & McGee, J. : Interim Report, N S.I. Contract No. CPE 80—15591 (1982).

7 Peterson, F C. : Wise L E. : U.S. Patent 2,192,202, Mar. 5, 1940.

8 Julien, L.M., Sun, B.C.H. Tappi 62 (8) : 63 (1979).

9 Kubes. G.J. : Trend (PPRIC) 29, 4 (1979).

10 Ketzen, R. : Fredrickson, R.E., et.al. pulp & paper 54 (8) : 144—149 (1980).

11 Kulkarni, H.G. et.al. IPPTA Silver Jubilee Seminar, New Delhi (1989).

12 Shenvi, A.K., Sirmokadam, N.N., Proc. VIII Carbohydrate Conf. 94, Nov (1992).

13 Murthv, N.V.S.R. et.al, IPPTA 3 (4) : 50–54, Dec. 1991