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JUTE RESEARCH AND DEVELOPMENT

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INDIA

Technical report: First mission report*

Prepared for the Government of India
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Pronoy K. Chatterjee
Expert in chemical softening of jute fibres

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Vienna

* This document has not been edited.

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SOFTENING OF JUTE BY CHEMICAL TREATMENT AT INDIAN JUTE INDUSTRIES' RESEARCH ASSOCIATION, CALCUTTA

REPORT ON FIRST MISSION
22ND OCTOBER - 19TH NOVEMBER 1988

ABSTRACT

Softening and upgrading of jute by chemical treatment, sponsored by UNDP was initiated in February 1988. This report describes a brief review of the status and recommendations on future planning. The report was prepared during the first mission for a period of October 22nd through November 19, 1988.

Laboratories for the project work are being constructed and the necessary laboratory equipment have been ordered. Excepting two entry level scientists and two supporting assistants, the team has been essentially formed for two UNDP programs in Applied Chemistry Division, viz. Chemical Softening, Bleaching and Dyeing of Jute Fiber.

Regarding the past work, IJIRA has already developed and commercialized through private industries a chemical softening agent, which is a partially sulfonated castoroil system and a low cost sizing material viz. Tamarind Kernel Powder (TKP). Referring to the chemical softening agent, the treatment with sulfonated castoroil system needs to be further optimized. Additionally, IJIRA's fundamental research on jute fiber morphology and other chemical treatments will provide supporting information for the current program.

Extensive technical interactions were carried out during the entire period with the Director and Heads of different divisions as well as project coordinators and other scientific personnel of IJIRA and The Reliance Jute Mill. Based on these interactions, a set of recommendations has been made for the expansion of the scope of the project.

RECOMMENDATION FOR FUTURE WORK

The following are the highlights of the recommendation:

1. Pilot plant work on optimization of sulfonated castoroil system as softening agent should be carried out through a standard statistical experimental design (Factorial design). This work should be expanded to optimize the conditioning of the treated fiber prior to carding.

2. Exploratory laboratory research should be carried out on treating the jute with <u>microemulsions</u> (amino silicone softener), which can be either obtained from a commercially available source or prepared in the laboratory by applying ultrasonic emulsifying technology or other appropriate techniques.

3. Fundamental research on porous structure of jute and solubility parameters of lignocellulose complex should be initiated for selecting optimum microemulsions for rapid and effective softening.

4. Application of selected debonding agents, viz. quarternary ammonium compounds (Berocel and Arquad), should be explored to soften the jute with minimal changes of fiber surface property. The quantity and application media need to be optimized.

5. Exploratory research on microwave heating of partially moist jute fiber might lead to a technique of controlled fracturing of the middle lamella which would soften jute with minimal strength loss. Incorporation of heat decomposible chemical prior to microwave heating should be alsc explored.

6. Possible adaptation of Chemical-Thermomechanical Pulping (CTMP) technology should be explored for controlled fracturing of middle lamella to soften jute. Controlled delignification technology should also be investigated.

7. To expand the scope of the current program and to explore new application potential of jute fiber, the following areas are suggested:

   a. Polymer grafting and substitution reaction as well as repellent coating techniques with fluorocarbon or silicon emulsion in order to convert jute to oil absorbent, water absorbent (super absorbent), water repellant material and elastic fiber.

   b. The feasibility of substituting bleached jute fiber in place of, or in combination with, wood pulp in disposable absorbent products.

8. Evaluation techniques for modified fibers should be further strengthened by developing test methods for thermal analysis, softness, resilience, fluid absorbency and retention and wet expansion and/or collapse.

9. In order to adapt and achieve maximum benefits from different technologies, a comprehensive literature review on fabric softeners, visit of IJIRA's senior staff members to selected foreign institutions involved in modern pulping technology including CTMP and textile institutions involved in fabric softening agents are recommended. The suggested institutions are:

   Swedish Pulp and Paper Research Institute
   Stockholm, Sweden

   Empire State Pulp & Paper Research Institute
   Syracuse, NY, U.S.A.

   Southern Regional Research Center, U.S.D.A.
   New Orleans, LA, U.S.A.

INTRODUCTION

Although jute is being used for high value decorative products, the traditional and bulk usage of jute is in the manufacturing of hessian burlap, sacking and carpet backing. Owing to stiff competition from synthetic fibers and plastics, the use of jute for those products has been gradually decreasing. Therefore, Indian Jute Industries' Research Association (IJIRA) has embarked on a comprehensive technical program to (1) upgrading the regular jute fiber by introducing certain unique properties to it including improved functional as well as aesthetic characteristics for high value products, and (2) upgrading the low quality jute through an economically viable chemical, physical and/or biological processes for its effective usage. In order to accomplish this task, IJIRA obtained financial and some technical assistance from the United Nations Development Program (UNDP). As per agreement, United Nations Industrial Development Organization (UNIDO) assumed the responsibility of executing the project activities in collaboration with the Government of India and IJIRA. The overall project is headed by the Director of IJIRA, Dr. S. R. Ranganathan, who is also acting as the National Project Director. Dr. Ranganathan's selected senior staff members are involved in implementing, coordinating and directing the technical aspects of the program. UNDP has appointed a technical coordinator for the entire project - Mr. R. R. Atkinson who is a private consultant and has an extensive background on jute processing technology.

The near term goals for the current UNDP programs are:

- Softening and upgrading of low quality jute.
- Improving productivity through the application of industrial engineering techniques.
- Cost reduction by redesigning of existing products.
- On-line instrumentation and process control.
- Development of new jute based products.
- Blends of jute and other fibers.
- Jute reinforced plastics.

Of these items, softening and upgrading of low quality jute by chemical means is one of the immediate goals of the project where research is being conducted under the direction of Dr. A. K. Mukherjee, head of the Applied Chemistry Division. In addition to the softening and upgrading of jute program, Dr. Mukherjee is also responsible for UNDP's project on bleaching and dyeing technology. The staff assigned for those two UNDP projects consists of 1 chemist, 1 assistant chemist and 1 lab attendant for the laboratory work; and 2 senior chemists (one for each project), 2 assistant chemists and 1 attendant for the pilot plant work. Currently, 2 assistant chemists and 2 attendants are in the process of being hired.

The laboratory facility for UNDP project has not been completed as yet. At present, the laboratory work is being carried out using the existing facilities of IJIRA. Among the laboratory equipment, most of the essential items are available in the Applied Chemistry Division except two vital pieces of equipment, TGA & DTA, which are being ordered. The project work began in August 1987 and the pilot plant work started in February 1988.

In order to reinforce the activities of the project on chemical softening of fibers, UNIDO has appointed the writer as a consultant for 3 split missions. The first mission has been conducted during the period of October 22 through November 19, 1988. This report summarizes the objective and the activities of the first mission.

OBJECTIVE OF THE PROJECT

"To develop chemical processes for softening hard jute cuttings and upgrading the quality of regular jute fiber."

In the past, continuous attempts were made to find a way of softening hard, rooty jute, to allow higher quantities to be used, thus reducing the total quantity of raw material for the mills. However, no successful techniques have been found until very recently when IJIRA introduced some chemical processes and some biochemical techniques which are gradually gaining industrial acceptance. This project is intended to intensify and expand the technology related to softening of the jute by chemical means.

ROLE OF CONSULTANT

As specified in the third mission report of Mr. R. Atkinson, the technical coordinator of the UNDP program, the following are the tasks of the consultant:

1. Review the work carried out by IJIRA on the softening and upgrading of low quality jute by the use of appropriate chemicals and suggest ways in which it could be improved.

2. Assist the IJIRA staff to implement pilot scale use of such chemicals.

3. Guide the IJIRA staff in ways of widening the scope for chemical softening in the industry.

4. Assist IJIRA staff to prepare a report in a form suited to the needs of the industry to encourage the use of chemical softening agents showing the technical and economic advantages to be gained by such practices. Also provide suggestions for future work program.

REVIEW OF IJIRA'S PAST WORK ON CHEMISTRY AND CHEMICAL TREATMENT OF JUTE

In 1986 IJIRA introduced a chemical, partially sulfonated castoroil, to soften the jute by incorporating it in the water-oil emulsion which is used in treating the jute fiber prior to carding. The chemical is sold commercially under the following trade names: Acenol, Jute Soft and Softener A-50. The process for applying sulfonated castoroil to jute fiber has been patented which is also licensed to many companies who have commercialized the technique. Even though a specific softening process has been developed, it has not been optimized to the extent where maximum technical and economic advantages are realized. The primary reasons for the selection of castoroil are its availability in large quantities and its non-edible feature.

IJIRA has also initiated a program to reduce the quantity of mineral oil required for treating jute, known as Jute Batching Oil (JBO). This is being attempted by mixing with JBO another proprietory product called Turkey Red Oil which is used in very, very small quantities. If this objective is accomplished, then industries will be able to save cost and the Nation will be able to save the foreign exchange by reducing the import of JBO.

Although not directly related to this project, IJIRA has also developed an inexpensive sizing agent for jute which is called Tamarind Kernel Powder (TKP). This treatment helps to smoothen the surface of fibers and thus facilitates the weaving process. They are also currently developing a guar gum sizing process. Because guar gum will produce the same sizing effect as TKP but with a lesser quantity, there is a further potential for cost savings. Also, unlike TKP, guar gum dissolves in cold water resulting in further savings in energy cost.

Regarding other efforts in improving the quality of jute, IJIRA has studied quite extensively the crosslinking of jute fiber using the traditional chemicals used for crosslinking of cellulose fibers. A technical publication and a D.Phil dissertation describes the process in detail. As expected, the crosslinking reaction produced crease resistance but lowered the tensile properties of fibers.

The retting of jute fibers is currently conducted by means of natural biological process which requires approximately 3 weeks time. An exploratory work is being conducted to accelerate the retting process by means of a chemical which reduced the time from 3 weeks to 2 hours. Also by application of the same chemical treatment, the rooty end opens up in fibrous form very easily. However, this work is at a very early stage and no publication is available at this time. Again, this development is not directly related to the project discussed in this report. It should be noted that a series of publications by IJIRA on lignin-carbohydrate linkages are quite relevant to the development of chemical retting technology.

A recent publication from IJIRA dealing with the morphological study of jute fiber with progressive delignification steps using scanning electron microscopy (SEM) provides insight information of the lignocellulosic structure which would be extremely useful for the project on chemical softening of jute. Also a series of publications of IJIRA on identification of various linkages of lignin and carbohydrates are quite important in terms of understanding the mechanism of the fiber stiffness and identification of chemicals required for softening.

Many other in-house and outside publications and reports are available which contain some relevant information concerning softening of jute, but they are not as such directly aimed toward the present objective. IJIRA's program on bleaching and dyeing is also a part of the UNDP program which is aimed toward producing aesthetically improved product and/or new usage of the product. Bleaching is being carried out by hypochlorite or hydrogen peroxide or both. Usually hypochlorite bleaching process removes the lignin from the surface whereas hydrogen peroxide alters the chromophores. Hydrogen peroxide bleach has a tendency to have brightness reversion under heat and/or ultra violet exposure. An IJIRA scientist has developed a buffer system to stabilize hydrogen peroxide; thus it produces reproducible results with different batches of samples. This work is underway.

IJIRA is looking into the feasibility of isolation and conversion of the components of jute into useful new products. Jute contains approximately 60% cellulose, 21-24% hemicellulose (including 5-7% xylan) and 11-14% lignin. Xylan can be hydrolized to xylose which is being used as a sweetener for diabetic patients. It can also be converted to furfural which is used in the plastic industry. Lignin can be converted to an adhesive or a binder material. Sulfonated lignin can be converted as a lubricant for the oil industry. Partially degraded cellulose residue after the removal of hemicellulose and lignin can be derivatized to make a soluble thickening agent. At present, the work is underway on the isolation of xylan and its conversion to xylose.

VISIT TO RELIANCE JUTE INDUSTRIES

Dr. A. K. Mukherjee and the writer visited Reliance Jute Industries at Kankinara, 24 Parganas, where Mr. T. C. Shah (General Manager), Mr. Bhandari (Manager of Standard Quality Control or SQC) and Mr. Maskara (Staff Engineer responsible for jute "softening" section) guided the tour to their manufacturing facility. Pat and Mesta of grades 3 to 7 are processed in the plant for manufacturing yarn, sacks and rolled fabrics. The softening of jute is carried out with the following formulation: mineral oil, water, non-ionic emulsifier (P-40) and "Jute Soft" developed by IJIRA. Reliance also uses TKP, another development of IJIRA, as a sizing material. The production capacity of Reliance is 125 tons/day for 3 shift operations.

IJIRA's pilot plant work on jute softening was conducted at Reliance. Further work on improving the distribution of the treatment during the storage prior to carding operation has been planned in the same facility. The plant is fairly clean and run in an efficient manner. Staff cooperation is good which is an essential attribute for conducting the pilot plant work in a production environment. However, certain safety features at the plant need to be improved in order to avoid the accidents and injuries.

This mill has been selected for commercial implementation of the pilot plant scale-up work for UNDP's chemical softening project.

DISCUSSION ON RECOMMENDED FUTURE PLANS

The chemical softening of the jute program can be divided into two categories:

1. Optimization of the sulfonated castoroil treatment at Reliance Jute Mill and expansion of the work with other treatment using commercially available chemicals similar to castoroil.

2. Exploratory and fundamental research to innovate other concepts to softening of jute as well as other chemical treatments to impart new properties to jute.

Pilot Plant Work on Optimization of the Sulfonated Castoroil System

As previously identified, either IJIRA pilot plant or Reliance Jute Mill could be used to optimize the treatment. Even though sulfonated castoroil has been commercially used in softening jute, it needs to be optimized for its formulation, application quantity, kinetics and uniformity to obtain maximum benefits with different types of jute materials such as low grade, high grade, end cuttings, etc.

The best and most effective way to optimize the individual chemical softener with least number of experiments is to conduct a set of statistically designed experiments, e.g., factorial design and then plotting and interpreting the response data. Such an approach would quickly identify the main variables and primary and secondary interactions of chemicals and the fiber structure. Subsequent experiments can then be designed to fine tuning the optimum conditions.

Once the experimental system is established and optimized for one type of chemical, other chemicals of the same homologous series could be routinely evaluated.

Additionally, the size and the condition of the storage of the treated jute need to be optimized first in the IJIRA pilot plant and then in Reliance Jute Mill using the statistical techniques.

Fundamental Research to Investigate Internal Plasticizers

Fundamental and basic research toward softening and upgrading of jute should consist of dual approaches: mechanistic as well as trial and error method.

In a mechanistic approach, basic knowledge on chemistry and morphology of jute fibers is essential. The study of chemical interactions of selected reagents with lignocelluloses and the porosity of the fiber would lead to identify the possible means to internally plasticize the fiber and thus enhance the softness and spinnability of fibers. It appears that the treatment with castoroil system, developed by IJIRA, softens the jute by penetration of the emulsion into the porous lignocellulosic structure and thus acts as an internal plasticizer.

In order to select other possible chemicals, if the solubility parameters of lignin and carbohydrate system are determined, the solubility parameter of chemicals could be matched by referring to chemical or polymer handbooks and thus a series of softening agents can be chosen for experimentation. During the experimentation, a significant amount of trial and error methods have to be followed.

Microemulsions

The softening chemical has to form a stable emulsion in order to achieve a practical method for treatment. It is important that the cell size of the emulsion is small enough to rapidly penetrate into the porous structure of fiber and interact with the gummy carbohydrate and lignin molecules. Thus, new amino silicon microemulsion softener developed recently by Dow Corning should be evaluated (Reference U.S. Patent 4,620,878). If this emulsion works, then a similar type of emulsion can be prepared with local chemicals. Various technologies to control the particle size of the emulsion should be attempted including ultrasonic treatment and other emulsification techniques.

Debonding Agents

Quarternary ammonium compound treatment in general would soften the jute. However, at a high dosage the fiber may slide on one another due to the reduced surface friction. But two specific types of quarternary ammonium compounds viz. Arquad and Berocel, commonly known as debonding agents, have been successfully applied in pulp technology where pulp fibers were softened without significantly altering its surface characteristics. Optimum quantities of these quarternary ammonium compounds may accomplish the present goal.

Microwave Heating of Jute Fiber

By proper selection of the frequency of microwave heating, it is possible to rapidly heat the moisture and thus explode the jute fiber. Such an explosion would fracture the middle lamella of the jute fiber and thus soften it. By optimizing the technique, it is possible that one can obtain the desired softness with minimal fiber strength loss. If simple heating of moisture does not work, some heat decomposible chemicals used in foaming technology may be introduced into the fiber prior to applying the microwave heating. It should be noted that the optimum frequency of microwave heating is very important. It may require quite a bit of experimental work before any sign of success can be seen.

Application of Chemical Thermomechanical Pulping (CTMP) and Chemical Delignification Processes

The structure of jute fiber has close similarity with the structure of hard wood. The fiber cells in hard wood can be separated by fracturing the middle lamella by a technique practiced for CTMP production where special equipment is used to apply pressure with steam followed by abrupt depressurization. In many instances the wood is pretreated with a chemical, e.g. sodium sulfite, for partially swelling the lignin. Such a process could be adapted to refine and soften the jute fiber. The process, of course, needs to be optimized to refine fibers to achieve the adequate softness with minimal strength loss. Some Swedish pulp and paper companies and the Swedish Pulp and Paper Institute in Stockholm could be visited to review the CTMP technology in more detail and then a pilot plant facility should be identified.

Also, in an appropriate outside location, the controlled delignification of jute fiber could be explored where the fibers could be refined to a specified degree without significant strength loss. Either sulfite or Kraft or a combination of both could be attempted using a continuous screw fed digestor. Such a process is being used for pulping sisal and other natural fibers.

Expansion of the Program to Develop Oil and Water Absorbent Jute Fibers as well as Water Repellant Fibers

Oil absorbent jute could be used for developing industrial wipes or cleaning fabric. Such a material can be produced by grafting vinyl monomers to jute fiber and/or by substitution and crosslinking reactions. Similar approaches with different monomers could be used to make water absorbent (superabsorbent) jute fiber which has a lot of potential applications in various health care and in disposable consumer products.

Polymer grafting technique can also be used for improving rot resistance and repellancy. Highly repellant jute fabric could be used for manufacturing tents, raincoats, etc. Many variations in properties of jute could be achieved by grafting techniques with the proper choice of monomers. The grafting reaction can be carried by using chemical initiators, gamma radiation or electron beam irradiation.

Water repellant treatment of fabric for raincoats, tents or for any other products where moisture proofing with breathable characteristics is required can be accomplished by optimizing the porosity of the fabric and subsequently treating with fluorochemical compounds, silicon emulsion or by urethane coating technique.

Use of Jute Pulp in Place of Wood Pulp

In many health care and disposable consumer products, bleached wood pulp is used. Because of the high cost of wood pulp, a large portion of which is imported, there is a need for local low cost natural fiber. It is conceivable that bleached jute pulp can be partially substituted in those products, provided the cost of jute pulp fiber is cheaper than the wood pulp.

Further Development of Characterization Methods

In addition to the current equipment, the IJIRA laboratory should be equipped with the following additional equipment and techniques:

1. Differential Thermal Analyzer and Thermogravimetric Analyzer (DTA & TGA) for determining thermal properties of jute including glass transition (Tg) of lignocellulosic complex for understanding the softening mechanism.

2. Fiber softness and resilience measurement techniques.

3. Equipment and methods for evaluating liquid absorption and retention of single fiber as well as fiber assembly, including their collapsibility and expansion characteristics on interacting with liquid.

4. A protocol for evaluating fiber softness by human panelists. This would require the establishment of a standard ranking system with proper questionnaire and an appropriate statistical analytical technique.

RELATED TECHNOLOGY DEVELOPMENTS IN OTHER ORGANIZATIONS

A brief review of the technical literature reflects that several other institutions are involved in various aspects of the development of staple and pulp fibers from jute by chemical means. Information available from their research programs in many ways would be quite valuable to the present project. It is not expected that all those programs would have a direct relationship to this project, but many subtle information or experimental methodologies could be adopted for the investigation of the softening of jute fiber.

Jute has a composition in many respects similar to wood pulp which is also a composite of cellulose, lignin and carbohydrates. The morphological feature of jute may be different from wood but the chemistry is close. The treatments of wood to partially isolate cellulose or separate the lignocellulose cells are being intensively studied in wood pulp industries and institutes. The information available from these studies should be reviewed in order to assess the potential application of certain chemical processes to alter the physical and chemical structure of lignin which may provide certain clues on how to soften the lignocellulosic phase of the jute. Particularly, a relatively new innovation in wood pulp industries known as Chemical Thermomechanical Pulping teaches how to fracture the lignin to produce soft fibrous materials from wood with 95% yield. This process in some form could be investigated for refining raw jute fiber to produce fine fibers. The technology of mechanical refining of pulp is another example which has been long practiced by pulp and paper industries.

In general, the countries which are leading the wood pulp technology are: Scandinavian countries, USA, Canada and Brazil, where Scandinavian countries, particularly Sweden, ranks the first.

In general, Brazil leads the non-wood pulp fiber technology where fibers are isolated from various types of plants, e.g., sisal, bamboo, bagasse, palm tree, banana stem, etc. United States' Department of Agriculture jointly with a U.S. company have developed a commercial process for Kenaf pulp. These technologies must be thoroughly reviewed in order to search for the techniques and fundamental information that are adaptable to the current program.

Last, but not the least, a thorough review of the technical literature on fabric softeners in general is highly desirable. Also, the fundamental research information on such areas as the penetration, porosity, swelling, wettability, relationship between emulsion droplet sizes and porous structure, solubility parameter of lignin and carbohydrate, etc. have to be developed in order to more effectively design an experimental program for a more machinistic approach in solving the problem rather than an empirical approach.

To start with, it is recommended that the relevant information be obtained on specific research programs that are continuing in the following institutions or industries:

1. Swedish Pulp and Paper Research Institute,
   Stockholm, Sweden

2. Empire State Pulp and Paper Research Institute
   Syracuse, New York, U.S.A.

3. Southern Regional Research Center,
   U.S.D.A., New Orleans, U.S.A.

It is suggested that some senior staff of IJIRA visit these organizations and review their current program and past research. The contacts with those institutes can be established through the present consultant for the visit of IJIRA staff.

FOLLOWUPS BY CONSULTANT

1. It has been agreed that the present consultant will attempt to obtain sample quantities of some softening agents recommended in this report including more detail information and a sample of amino silicon microemulsion which has been recently disclosed in a USA patent.

2. Information on the development and modification of natural fibers available internationally will be periodically forwarded to IJIRA.

3. Technical progress reports on the project work prepared by IJIRA scientists will be reviewed and comments will be forwarded on requests from the division head or the director of IJIRA.

4. Visit for the next mission will be scheduled for June/July 1989.