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Research Paper

Ankara (Calotropis Procera) – As a source of Eco-friendly alternate Lignocellulosic Raw Material for Handmade Paper:

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ABSTRACT
In India, Handmade paper industry has been identified as one of the village industry and it has witnessed significant growth in recent past because of improved demand of handmade paper not only on national level but also in the international arena.
The Industry is highly fragmented varying from a very tiny sized unit (average 100 Kgs per day.) to reasonably sized unit (more than 2 tonnes per day). Though the industry has achieved remarkable growth however, in the recent past the industry has been struggling for its sustenance due to various reasons. The most prominent reason encountered is the scarce availability and continuously increasing price of the cotton hosiery waste, one of the commonly and traditionally used raw material for handmade paper making. The raw materials selected namely ***Ankara (Calotropis Procera)*** *to establish it's suitability as cellulosic raw material for making pulps for handmade paper industry. This should help in providing a cost effective, good quality cellulosic raw material as an alternate to cost prohibitive traditionally used cotton hosiery waste for manufacturing good quality handmade paper & its products.*
This will help in improving the cost economics & competitiveness of the Indian paper industry in the global market besides addressing the problems of environment & global warming

KEYWORDS
KNHPI- Kumarappa National Handmade Paper Institute,*GSM – Gram per square Meter,APA – Alkaline Pulping at Ambient Temperature, APB – Alkaline Pulping at Boiling Temperature, APP – Alkaline Peroxide Pulping, ASP – Alkaline Sulphite Pulping, cuen – Cupriethylenediamine, EDTA – Ethylene diamine tetra acetic acid disodium salt*

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I. INTRODUCTION:

For over 2000 years the craft of handmade papermaking has been practiced all over the world utilizing a variety of techniques. The review of literature suggests that the evolution of hand papermaking and its cultural significance changes with the evolution of mankind. Paper's evolution has been shaped by the structure and chemical composition of the fibres. Almost every aspect of modern papermaking technology has been foreshadowed by traditional practices. Such practices were passed down for many generations within families of papermakers. The main sources of cellulosic fibre evolved aesthetic ancient craft migrated from its birthplace to many other part of world. The basic raw material papyrus changes to other bast and leaf fibres in the world. Though most paper made today comes from automated, continuous production systems, handmade paper has enjoyed resurgence, both as a traditional craft and as an art-form. In addition, traditional papermaking methods can provide insights to help in modern applications involving cellulosic fibres.

The raw materials selected namely **Ankara (Calotropis Procera)** to establish it's suitability as cellulosic raw material for making pulps for handmade paper industry. This should help in providing a cost effective, good quality cellulosic raw material as an alternate to cost prohibitive traditionally used cotton hosiery waste for manufacturing good quality handmade paper & its products.

This will help in improving the cost economics & competitiveness of the Indian paper industry in the global market besides addressing the problems of environment & global warming.

The experimental study of the fibre structure and chemical composition including cellulose, lignin and extractives are based on investigation of Ankara fibres which were collected locally.

Photograph of the raw material and fibres structure:

ANKARA PLANT

ANKARA FIBRE

II. ANKARA CELLULOSIC RAW MATERIAL – FIBRE EXTRACTION METHOD AND YIELD.

It is calcareous plant which grows in sandy soil. Ankara belongs to genus Calotropis. This genus consists of about 6 species. Its shrubs or small trees distributed in tropical and subtropical countries of Africa and Asia.

It is available in Haryana, Punjab, Gujarat, Bihar and Rajasthan and widely available in North West. Rajasthan in the area of Bikaner, Jodhpur, Pali, Jhunjhunu, Jaisalmer and Barmer.

Three of the species occur in India and two of them are of economic importance namely *C.giganta and C.procera*. Both plants yield latex containing gutta-percha like cautchouc and contain a cardiac poison. It is a weed and yield strong fibres. In the present study C. procera is used.

Fibre Extraction Process

The locally collected fibres are extracted after retting (a microbial process that breaks the chemical bonds holding the stem together and thereby allowing separation of the bast fibres from the woody core) process. The bast is taken out by hammering or stamping. The epidermal green layer, the mucilage and the pithy stems are thus separated. The bast fibres of Ankara are strong and semi- white and consisting 2% of O.D. weight of the stem. The fibres of Ankara, also called Aak, are very strong in terms of physical strength properties.

Proximate Chemical Compositions of Ankara Fibres:

Particulars	Ankara fibre
Hot Water Solubility,%	07.00
N/10 NaOH Solubility,%	15.11
Alcohol-Benzene Solubility %	21.10
Ash, %	04.50
Lignin, %	07.50
Holocellulose,%	89.00
α-cellulose, %	81.10

Fibre Morphology:

The fibre morphological studies of ankara have been carried out. The fibre length and diameter which, considered to be an important factor from view point of paper making, were determined, and results recorded in table.

Table: Length and Diameter of Ankara Fibre:

Common name (Scientific name)	Fibre Length (mm)		Fibre Dia (μm)	
	Avg	Range	Avg	Range
Ankara (*Calotropis procera*)	3.20	2~20	23.00	16~40

III. PULPING OF THE RAW MATERIAL USING ECO-FRIENDLY CONDITIONS:

The Indian Handmade Industries are said to be eco-friendly in nature and it is. Keeping the concept of eco-friendliness, the selected raw material was subjected to different methods of pulping and eco-friendly bleaching process by using mild chemicals treatment and other operating parameters. Studies carried out on delignification of these ligno-Cellulosic raw materials i.e. ankara by exploring different eco-friendly compatible pulping process as follows:

1. Alkaline Pulping at ambient temperature and atmospheric pressure.
2. Alkaline pulping at boiling temperature.
3. Alkaline peroxide pulping process (APP).
4. Alkaline sulphite pulping process (ASP).

Preparation of Handmade Paper Sheets from the Pulps Obtained from the Identified Raw Materials:

The ankara pulped raw materials, after beating at desired freeness level i.e. CSF (Canadian Standard Freeness) subjected to handmade paper sheet making as per the standard methods.

Evaluation and Testing of Handmade Paper Sheets for Different Properties of interest viz. Tensile Index, Tear Index, Burst Index, Double fold and Brightness:

After sheet making, these would be dried, conditioned and will be got tested for tensile, tear, burst, double fold and brightness as per the standard methods of testing.

Evaluation of Physical and Optical Strength of Pulp Sheets:

The papers thus produced from the identified raw material from different pulping conditions will be evaluated for various parameters as follows:-

Strength & Optical Properties:

- Tensile Index, (Nm/g).
- Tear Index, (mN. m^2/g).
- Burst Index, (KPa. m^2/g).
- Double Fold, (No.)
- Brightness, (% ISO).

III. 1. Alkaline Pulping at Ambient Temperature and Atmospheric Pressure (APA) – Pulp - Yield, Strength Properties of Pulp Sheets:

In this process of pulping the identified raw materials were soaked with 8%, 10% and 12% of NaOH for 24Hrs, as per the pulping conditions.

Washing: The cooked pulps after the pulping were transferred to a fine terylene cloth and the surplus black liquor is squeezed out for effluent characterizations& washed well with the fresh water. Pulp yields for the fibres were evaluated.

Pulp Evaluation: The cooked pulps were run in to laboratory valley beater to achieve the desired CSF (Canadian Standard Freeness) value i.e. 300 ml. From these beaten pulps, hand sheet on British Sheet making machine were made as per the standard procedure and dried them.

After conditioning, the physical and optical strength properties were evaluated as per the Standard Test Methods viz TAPPI, BIS, IS & ISO:2471. The test results are shown in table- 1, show the physical and optical strength of ankara sheets obtained from pulping process at ambient temperature and atmospheric pressure.

Table: 1. Pulp - Yield, Physical and Optical strength properties of the pulp sheets from alkaline pulping at ambient temperature and atmospheric pressure process (At 300 ml CSF):

S.N.	Characteristics	Ankara		
	CSF,ml	**300**		
	NaOH %	8	10	12
	Pilp Yields, %	88.90	87.00	86.00
1	Tensile Index (Nm/g)	27.00	28.41	30.00
2	Tear Index ($mN.m^2/g$)	1.57	1.75	1.90
3	Burst Index ($Kpa.m^2/g$)	1.78	2.10	2.25
4	Double Fold, No.	1100	1350	1470
5	Brightness (%) ISO	35.50	34.75	35.20

Results and Discussions:

The results of pulp yields at various conditions are shown in table-1. From the results it could be observed that, the pulp yield of ankara (86.78%) at the pulping using 12% of NaOH. The results show that the acceptable quality of the pulp in respect of double fold which is one of the important property for high grade speciality paper, could be made. The double fold of Ankara is 1630.

This pulping method is suitable for such handmade paper industry which does not have the cooking facility like digester and are willing to use these identified raw materials.

The pulp produced by this method can be used for making variety of handmade papers / paper boards.

The pulps obtained from this pulping method were not suitable for making tissue paper / light gsm paper further.

III. 2. Alkaline Pulping at Boiling Temperature and Atmospheric Pressure (APB) – Pulp - Yield, Strength Properties of Pulp Sheets:

In this process of pulping the identified raw material ankara was treated with 8%, 10% and 12% of NaOH.

Washing:The cooked pulps after the pulping were transferred to a fine terylene cloth and the surplus black liquor is squeezed out for effluent characterizations& washed well with the fresh water. Pulp yields for the fibres were evaluated.

Pulp Evaluation: The cooked pulps were run in to laboratory valley beater to achieve the desired CSF (Canadian Standard Freeness) value i.e. 300 ml. From these beaten pulps, hand sheet on British Sheet making machine and Tissue Sheet making machine, were made as per the standard procedure and dried them.

After conditioning, the physical and optical strength properties were evaluated as per the Standard Test Methods viz TAPPI, BIS, IS & ISO:2471.The test results are shown in table-2, show the physical and optical strength of Ankara pulp sheets obtained from pulping at boiling temperature and atmospheric pressure (APB).

Table: 2 Pulp -Yield, Physical and Optical strength properties of the pulp sheets from alkaline pulping at boiling temperature and atmospheric pressure process (At 300 ml CSF):

S. N.	Characteristics	Ankara		
	CSF,ml	**300**		
	NaOH %	8	10	12
	Pulp Yield, %	85.05	84.00	83.11
1	Tensile Index (Nm/g)	68.00	69.80	70.57

2	Tear Index (mN. m^2/g)	4.40	4.70	4.50
3	Burst Index (Kpa.m^2/g)	3.60	4.00	4.57
4	Double Fold, No.	8200	8351	8510
5	Brightness (%) ISO	35.00	34.00	35.00

Results and Discussions:

From the results indicated in table-2, it could be observed that at NaOH concentration of 8% with reasonable high strength properties in respect of double fold tensile index tear index and burst index. The pulp yield in case of identified fibres i.e. ankara which could be considered a very good pulp yield from any lingo-cellulosic fibres. The inherent chemical composition of the identified fibre having lower lignin and higher cellulosic content could attributed to such high yield pulp. The data of the strength properties i.e. appreciably high double fold may be taken as the very good strength properties which could be reflected from the inherent fibre morphological character of this fibre i.e. the higher fibre lengths and fibre diameters.

This pulping method is suitable for such handmade paper industries which have the cooking facility like open digestion, where stationary / vomiting type digester is required for the pulping and wants to use these identified raw materials. In place of the vomiting type digester, open boiling in any big utensil could be preferred to curtail the expense of digester. During the pulping, proper bath ratio should be maintained time to time for proper cooking of the raw materials, which results in better delignification. The pulps, thus, produced can be used for making variety of handmade papers / paper boards. The physical strength properties of the fibre produced at boiling temperature and atmospheric pressure (APB) showed a remarkable change as compared to the pulping at ambient temperature and atmospheric pressure (APA).

III. 3. Alkaline Peroxide Pulping, (APP)– Pulp - Yield, Strength Properties of Pulp Sheets:

The alkaline peroxide pulping process is the further development of the earlier two pulping methods i.e. pulping at ambient temperature and atmospheric pressure & pulping at boiling temperature and atmospheric pressure, to address the problem of environment pollution and to retain eco-friendly credential of handmade paper making from the identified raw materials.

In this process of pulping the identified raw materials were treated with 8% NaOH + 2% H2O2, 10% NaOH + 2% H2O2and 12% NaOH + 2% H2O2, at 95°C. as per conditions. The temperature of 95°C was maintained properly to get better brightness results from hydrogen peroxide. Before pulping following pretreatment were followed to get better results.

Treatment with chelating agent: The plant materials ankara contains metal ions like iron, manganese etc. which, if present may decompose hydrogen peroxide and form complex with the metallic ions affecting the brightness and permanence of paper.

Therefore, to avoid the interference of metallic ions, pretreatment of raw material with chelating agent EDTA (Ethylene diamine tetra acetic acid disodium salt) was carried out. The following conditions were applied.

EDTA: 0.5%,
pH: 4 to 5
Time: 20-30 Mints
Temperature: Ambient temperature

After chelation, these raw material was cleaned by de-mineralized water to avoid the hindrance of metallic ions further, if present, the effect of hydrogen peroxide will not occurred.

Washing: The cooked pulps after the pulping were transferred to a fine terylene cloth and the surplus black liquor is squeezed out for effluent characterizations& washed well with fresh water. Pulp yield for the fibre was evaluated.

Pulp Evaluation: The cooked pulps were run in to laboratory valley beater to achieve the desired CSF (Canadian Standard Freeness) value i.e. 300 ml. From this beaten pulp, hand sheet on British Sheet making machine and Tissue Sheet making machine, were made as per the standard procedure and dried them.

After conditioning, the physical and optical strength properties were evaluated as per the Standard Test Methods viz TAPPI, BIS, IS &ISO: 2471.The test results are shown in table-3, show the physical and optical strength of Ankara sheets obtained from APP at 95°C temperature and 1.40 Kg/cm^2 of pressure (APP).

Table: 3. Pulp - Yield, Physical and Optical strength properties of the pulp sheets from alkaline peroxide pulping (APP) at 95°C temperature and pressure of 1.40 Kg/cm^2 process (At 300 ml CSF):

S. N.	Characteristics	Ankara		
	CSF, ml	300		
	NaOH,%	8	10	12
	H2O2, %	2	2	2
	Pulp Yield ,%	74.00	73.57	72.00
1	Tensile Index, (Nm/g)	78.11	80.00	81.50
2	Tear Index (mN. m^2/g)	4.70	5.00	5.20
3	Burst Index, (Kpa.m^2/g)	5.60	6.00	6.43
4	Double Fold ,No.	9880	9955	10001
5	Brightness (%) ISO	37.00	36.00	35.00

Results and Discussions:

The result of pulp yields and strength properties using APP process for banana, ankara and pineapple fibres at various process conditions i.e. 8%, 10% & 12%, with 2% H2O2 at a temperature 95°Cand maintaining bath ratio of 1:8 for 3 Hrs are shown in table-3.

From the results indicated in table-3, it could be observed that the pulp yields 75.00% for ankara. The strength properties were quite good in respect of all the parameter such as tensile index from. The strength properties like tear index and burst index were also quite high. The above strength properties indicate that the fibre could be utilized for manufacturing of high value added paper including the security, currency papers and could be a substitute or a source for its blending for the cotton hosiery fibre, which is being conventionally used for the security and currency papers.

The digester was used for the pulping of identified lignocellulosic raw materials which results better delignification. It is recommended to use digester and temperature should be maintained at 95ºC during the digestion to avoid the evaporation and decomposition of hydrogen peroxide.

Thus, this pulping method is very much suitable for such industries where the cooking facility like rotary spherical digester is used provided the industry is willing to use the identified fibrous raw materials.

III. 4. Alkaline Sulphite Pulping (ASP) – Pulp - Yield, Strength Properties of Pulp Sheets:

In order to achieve the zero discharge of black liquor resulting from pulping and in order to minimize the pollution load in the effluents, an efficient and new pulping was designed in alkaline sulphite pulping process. The identified raw material was treated with total chemical of 8% (70% NaOH + 30 % Na2SO3), 10% (70% NaOH + 30 % Na2SO3) and 12% (70% NaOH + 30 % Na2SO3) at 120°C as per the conditions. The temperature of 120°C was maintained properly to get better delignification.

Washing: The cooked pulps after the pulping were transferred to a fine terylene cloth and the surplus black liquor is squeezed out for effluent characterizations& washed well with fresh water. Pulp yield for the fibre were evaluated.

Pulp Evaluation: The cooked pulps were run in to laboratory valley beater to achieve the desired CSF (Canadian Standard Freeness) value i.e. 300 ml. From these beaten pulp, hand sheets on British Sheet making machine and Tissue Sheet making machine, were made as per the standard procedure and dried them.

After conditioning, the physical and optical strength properties were evaluated as per the Standard Test Methods viz TAPPI, BIS, IS &ISO: 2471.The test results are shown in table-4, showing the physical and optical strength of Ankara pulp sheets obtained from ASP process at 120°C temperature and 2.00 Kg/cm^2 of pressure.

Table: 4. Pulp - Yield, Physical and Optical strength properties of the pulp sheets from alkaline sulphite pulping (ASP) at 120°C temperature and pressure of 2.00 Kg/cm^2 process (At 300 ml CSF):

S. N.	Characteristics	Ankara		
	CSF, ml	300		
	Total Chemical, %	8	10	12
	NaOH,%	70	70	70
	Na2SO3, %	30	30	30
	Pulp Yield, %	73.00	72.00	70.50
1	Tensile Index, (Nm/g)	77.01	78.50	80.31
2	Tear Index, (mN. m^2/g)	4.50	5.57	6.75
3	Burst Index, (Kpa.m^2/g)	4.92	5.70	6.00
4	Double Fold,No.	8700	9000	9500
5	Brightness (%) ISO	38.00	37.00	36.75

Results and Discussions:

The pulp yield was found to be comparable with that of alkaline peroxide pulping process. The strength properties are also at par with the APP processed pulp sheets. However, brightness of the pulp of identified raw material was slightly less.

The digester was used for pulping of identified lignocellulosic raw materials which results in better delignification. It is recommended to use digester and temperature of 120ºC should be maintained during the digestion.

From the data in table-4, it is observed that the strength properties viz. tensile, tear, burst and double fold are increasing with the increase in total chemical dose i.e. NaOH &Na2SO3 for the identified raw material.

This pulping method is very much suitable for such handmade paper industries where the cooking facility like rotary spherical digester are used provided the industry is willing to use the identified fibrous raw materials.

The pulp produced can be used for making variety of handmade papers / paper boards as the physical strength properties of these fibres by ASP at 120°C temperature showed better results as compared to alkaline pulping (at ambient temperature and atmospheric pressure (i.e. APA) & boiling temperature and atmospheric pressure (i.e. APB) and at par with alkaline peroxide pulping (i.e. APP).

Further, the pulps obtained from this pulping method would be suitable for making tissue paper / light gsm paperwith high age property.

The one of the major advantage of using ASP process is that the black liquor generated in pulping could find an application, as a source of lignosulphonate, which is being used in concrete admixture as retarder.

IV. SUMMARY:

Handmade Paper from the Alternate Lignocellulosic Raw Materials viz. Ankara Fibre:

1. Conventionally, the raw materials used most commonly in handmade industry are the off cut of the textile industry i.e. hosiery waste in white colour as well as in mixed colors, cotton rags, off cut from HMPI, and recycled secondary fibres but the availability of these raw materials for handmade paper industry is becoming scarce and very expensive. As a matter of fact, the existence of handmade paper industry has become difficult and more than 50% of the units have been closed. This has resulted in decreased production and adversely affected on the exports & domestic market besides decreased opportunities for employment generation. Looking into the problem of availability of cellulosic raw material for handmade paper industry and to make the raw material available at cheaper and affordable price. There is an urgent need to identify alternate source of locally and an easily pulpable raw materials, which could be made available for handmade paper industry at cheaper price.

2. Ankara being the bast fibre contains rich amount of cellulose which can be exploited for manufacturing of good quality handmade paper and products.

3. Studies on the fibre morphology and chemical nature of identified raw materials indicated their suitability for manufacturing of high quality handmade paper and paper products like **Calligraphy paper, Writing Printing Paper as well as File Cover, Card Sheets, Leather, Moon Rock, Silk Paper, Batik Paper ect and many more.**

4. While comparing the chemical composition with conventionally used cotton hosiery waste, the identified lignocellulosic raw materials were found to contain around 80% of α-cellulose contents, lower content of lignin (less than 12%), which are considered to be one of the important parameter for their suitability for handmade paper making. Further, the fibre lengths and diameters were found to be appreciably higher (length more than 3 mm and dia more than 24 μm) in all the identified fibres which were comparable or higher than the conventionally used cotton hosiery waste.

5. Due to the inherent chemical nature of the identified ligno-cellulosic raw materials viz. Ankara required use of milder doses of NaOH, hydrogen peroxide and Sodium sulphite to delignify or dissolve lignin and bleaching of the cellulose rich pulp to make it suitable for handmade papermaking. Thus, the efforts were made to make the delignification and / or bleaching process an ecofriendly process to retain ecofriendly credentials.

6. The research work focused on using eco-friendly pulping and bleaching process, viz. Alkaline Pulping, Alkaline Peroxide Pulping & Alkaline Sulphite Pulping processes using milder conditions in respect of use of chemicals, temperature during the pulping and bleaching of the identified lingo-cellulosic raw materials to produce pulps, suitable for handmade papermaking.

7. Looking into the high quality pulp thus, produced from the identified fibrous raw materials under the optimized process conditions, it has provided the scope for blending of these fibres for partial replacement of cotton hosiery waste to produce handmade paper of special variety with possibility to produce high end products like currency papers and security papers.

8. Moreover, the resultant pulp produced for ankara having better durability and double fold with longer fibre length could make possible to produce fine quality of tissue papers in various grammage, which found an application in archival purpose for **preservation and conservation of old documents and manuscripts**, which provide greater value to the paper and paper products produced from this identified fibrous materials.

9. The present research work, thus, should prove to be of an immense advantage to provide an alternate and good quality fibre source available as waste bio-mass in different part of the country. This will help not only in providing a cheaper source of raw materials for the very survival of the sick units, which are at the verge of closure, but also address the problem of environmental problem which otherwise is caused due to dumping of these ligno-cellulosic mass as a waste.

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