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Isolation and Characterisation of Agave Cantala (Dakatia) Fibre

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Abstract *Agave Cantala (Dakatia) fibre is a lignocellulose fibres which grows in Bangladesh without care here and there. It is a leaf fibre. The fibre contains 70% α -cellulose, 16.6% hemicellulose, 7% lignin, 1.3% Pectic material, 0.5%, aqueous extract, 2% fatty and waxy materials and 2.6% miscellaneous. The higher amount of cellulose shows the higher crystalline polymer which affects its overall performance. The molecular weight of Agave Cantala fibre (α-cellulose) was taken by viscometric method. The average molecular weight of α-cellulose for raw Agave Cantala fibre was found to be 134831 and bleached Agave Cantala fibre was found to be 126404 which is reduced at high temperature.*

Introduction

Agave is a Philippine plant yielding a hard fibre used in making coarse twine. A cordage fibre obtained from the leaves of a tropical plant, *Agave Cantala.* The plant itself also called maguey. *Agave Cantala* is one kind of sisal fibre[11]. Its local name is DAKATIA TREE.

Maguey, scientifically known as Agave Cantala, of the amaryllidaceae, family originated from maxico and to some extent cultivated in the Philippines specifically in Central Visayas and Northern Luzon. In Bangladesh maguey plant grows in Chuadanga, Jessore[1] and the frontier line of West Bengal.

Agave Cantala is a native of the Yucatan Peninsula, Mexico[12]. *Agave Cantala* grows best in a hot climate and may be grown throughout the humid and sub-humid lowland tropics. *Agave Cantala* is a squat plant with long, knife shaped leaves that form a rosette close to the ground. These fleshy, rigid leaves, from which the Agave fiber is derived, are usually grayish-green to dark green. The fiber within is coarse, long and extremely strong shiny and attractive, its color is usually creamy white.

Fiber may be found in almost any point of the plant stems, leaves, roots, fruits and ever seeds. The four chief types classified according to their origin include best fiber wood fiber sclerenchyma cells associated with the vascular bundle strands in leaves and surface fiber which are hair like out growths on the seeds of various plants. The use of the "leaf fibers" is often to criticism on the ground that it gives on indication as to the particular tissue or origin in which the fibers occur, vegetable fiber are obtained from nature as single and multiple cellular systems. *Agave*

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Cantala has a multiple cell systems. The ultimate in the aggregate bundle are bound together by natural polymers variously called resins gums cementing materials encrusting materials.

A maguey plant grows into a rosette of leaves which drops to a horizontal position when it matures. A rosette usually consist of 20 to 50 leaves. The leaves which crowd on the stem are fleshy, thick and persistent for several years. This grayish leaves end in terminal spines bearing marginal pickles[1]. Each leaf is 5ft long while its width ranges from 2.5 to 4 inches and has an average weight of 200 gm.

Agave Cantala is a perennial succulent which, with good growing conditions forms an inflorescence after 6 - 9 years after having produced 200 - 250 leaves, and then dies. Leaves average 120cm in length and are arranged spirally around the thick stem. The leaves are 75% schlerenchyma bundles. The root system is shallow but extends up to 3.5m from the stem.

Maguey *(Agave Cantala)* grows on arid soils worldwide, with superior tear and tensile strength compared to wood. Because it does not displace important crops and can switch from paper to hard fiber cordage depending on price, its bright white sheets (requiring little bleach) make it a prim candidate for a pine/sisal combo paper.

Agave Cantala is essentially a commercial crop hardly ever grown by small-scale farmers except as hedges. The greatest demand for *Agave Cantala* is for use as binder twine, but it can also be used to make ropes, sacks and bags of various types as well as marine cordage. *Agave Cantala* is a strong, stable and versatile material that can be woven into boucles and rib weaves, flat weaves, and jacquard patterns and many dyed colors. It can be a little rough underfoot, but so when combined with other fibres such as wool. *Agave Cantala* carpets are naturally sound-absorbing, anti-static, and extremely durable because of the inherent qualities of this tough, hard-wearing fibre. It is also naturally insulating and difficult to ignite. *Agave Cantala* absorbs moisture readily.

Agave Cantala occupies 6th place among fibre plants, representing 2% of the world's production of plant fibres (plant fibres provide 65% of the world's fibres) [5]. The world's largest producers are Brazil (199,000t), Kenya (40,000t), Tanzania (28,000t) and Madagascar (20,000t). There are significant exports only from Brazil (65,000t), Kenya (31,000t), Tanzania (18,000t) and Madagascar (9,000t).

Agave Cantala fibre has Length-100cm, Diameter -0.1 to 0.46 mm, Fineness-9 to 409 denier, Cell length-0.8 to 8 um, Cell width-7 to 47 m, Shape-Cylindrical, Porosity-17, Apparent viscosity-1.20 g/cc, True viscosity-1.45 g/cc, Volume resistivity-0.47 to 0.5 X105$^{\Omega}$cm, Dielectric strength - 5KV [7].

Fibre extracted from the leaves of the *Agave Cantala plant* and its hybrids can be used to produce high quality papermaking pulp. Agave Cantala pulp has certain characteristics such as high tear resistance, high alpha cellulose content, high porosity, high bulk, high absorbency and high folding endurance which make sisal pulp suitable for many specialty papers. Also, because sisal pulp has physical properties superior to softwood Kraft pulp, there may be opportunities to cost effectively replace softwood Kraft with *Agave Cantala* pulp in commodity papers. For example, *Agave Cantala* pulp may be used as a reinforcing fibre in high recycle content papers, or its use may permit basis weight reductions while maintaining product quality. Markets for *Agave Cantala* pulp are established in the specialty paper sector; however, currently there are no markets established in the commodity paper sector. Before considering potential market opportunities for *Agave Cantala* fibre in the pulp and paper industry, we must first consider the fibres currently in use and certain trends that may favor using *Agave Cantala* fibre in the future [9, 6, 5, 2].

Materials and methods

Isolation of the constituents of Agave Cantala fibre:

Agave Cantala fiber collected from the local area (Chuadanga, Jessore, Kushtia). Bottom portion was taken for investigation. Agave Cantala fiber was scoured in a solution containing 6.5 gm of soap flake and 3.5 gm of soda per liter at 75 °C for 30 minute in a large beaker. The ratio of the fiber to solution was 1:50. The fiber was thoroughly washed with distilled water and dried in the open air [10, 3, 11].

Estimation of Aqueous extract:

Certain amount of *Agave Cantala* fiber dried at 105 °C, is heated with distilled water at 60 °C for two hours and the fiber is separated by filtration, dried at 105 °C for constant weight. The loss in weight gives the amount of aqueous extract in the fibers.

Estimation of fatty and waxy matters:

The *Agave Cantala* fiber at 105 °C is immersed in a benzene-alcohol mixture (2:1 by volume) contained in a beaker in the ratio 1 gm fiber per 100 ml of the mixture and then allowed to stand for 10 hours with occasional stirring. The Agave Cantala fibers are separated from the mixture by filtration, subsequently washed several times with fresh benzene alcohol mixture and finally with alcohol and then the fiber is dried at 105 °C for constant weight. The loss in weight on extraction with the solvent mixture gives the amount of fatty and waxy matters in the *Agave Cantala* fibers.

Estimation of pectic matters:

Among the several methods for the estimation of pectic matters, A. G. Narmen10 methods is the best method which is given below:

The Agave Cantala fiber dried at 105 °C is taken in a beaker and heated with a 0.5% ammonium oxalate solution in the ratio of 1 gm fiber per 100 ml solution at 70-80 °C for three days in a heating mantle. As evaporation goes on the loss of water is compensate by adding hot distilled water to keep the level of the solution constant throughout the process. The fiber is then filtered, washed thoroughly with hot distilled water then dried at 105 °C for constant weight. The loss in weight gives the amount of pectic matter in the *Agave Cantala* fiber.

Estimation of lignin:

Isolation of Lignin is achieved in two different ways:

1. Dissolution or destruction of the carbohydrates by means of suitable sol vents, such as 72% sulphuric acid, supersaturated hydrochloric acid (42%) or cuprammonium to level the lignin as a insoluble residue.

2. Dissolution of the lignin either in a more or less unchanged form or as a derivative. In both cases, removal of the components extractable with benzene-alcohol mixture (2:1) is essential to avoid contamination of the lignin.

Numerous method have been proposed for the quantitative determination of lignin in cellulosic materials, the most common being based upon the assumption that lignin remains as the un-saccharidable residue when the carbohydrate dissolves in strong mineral acids.

Dewaxed and depectic fibers dried at 105 °C treated with 72% sulphuric acid 15 ml for 1 gm of the fiber with frequently stirring at ordinary temperature. The mixture is allowed to stand for 2 hours and then diluted to 3% acid concentration. After refluxing the mixture for 4 hours it is allowed to stand for over night and filtered through a sintered glass funnel and washed thoroughly with hot distilled water. The constant weight of the residue in the sintered funnel dried at 105 °C gives the amount of the lignin content of the *Agave Cantala* fiber.

Estimation of α-cellulose and Hemicellulose:

In this step all non cellulosic matters in fiber are removed by the treatment of the bleaching agent, such as sodium chloride when chlorite holo-cellulose, a combination of α- cellulose and hemicellulose is obtained.

Preparation of chlorite Holo-cellulose:

A suitable amount of dewaxed and depectinised fiber dried at 105 °C is treated with

0.7% sodium chlorite solution buffered at 105 °C pH = 4 in the ration 1 gm fiber per 80 ml liquor at 90-95 °C for 90 minutes; 1 ml buffer solution of sodium acetate and acetic acid of pH = 4 is added for every 10 ml of chlorite solutio to stabilized the constant pH. The fiber is then filtered and washed thoroughly. The cellulosic materials obtained in the sintered funnel are called the chlorite holo-cellulose, which is dried at 105 °C till constant weight is obtained.

Separation of α-cellulose from Hemicellulose:

Chlorite holocellulose dried at 105 °C is treated with 24% KOH solution for 4-hours with occasional stirring the ratio 1 gm fibre per 100 ml of alkali solution. By this treatment hemicellulose goes into solution and cellulose remains un-dissolve. The α-cellulose is separated by filtration, washed throughly with 2% acetic acid solution, finally with distilled water and then dried at 105 °C for constant weight. The amount of α-cellulose thus obtained is deducted from the weight of holo-cellulose taken gives the amount of hemicellulose.

Methods of determination of molecular weight[4]

Viscosity measurements have been utilized more than any other type of measurements in attempting to derive information concerning dissolved macromolecules. This increase in viscosity is a function of the size, shape and concentration of the polymer molecule in solution. If the relation between these parameters and viscosity were known it would be possible to measure molecular weight and molecular dimension of the macromolecule by viscosity.

Agave Cantala (maguey) fiber is not purely cellulosic fiber. It contains α- cellulose (70%), hemicellulose (16.6%), lignin (7%), pectic matter (1.3%), aqueous (0.5%) fatty and waxy matter (2%), mineral matters and miscellaneous (2.6%). From the above composition we see that the main constituents of Agave Cantala (maguey) fiber is α-cellulose. So the molecular weight of Agave Cantala (maguey) fiber generally means the molecular weight is α- cellulose. For the determination of molecular weight of α-cellulose, at first it is necessary to remove all other constituents from the *Agave Cantala* (maguey) fiber by the usual method. There are two methods used for the determination of molecular weight of α- cellulose.

1. Indirect method:

In this method cellulose is first converted to a derivative by substitution of some of its hydroxyl group, such as nitro acetate as xanthate groups.

2. Direct method:

In this method mineral acid e.g. phosphoric acid is used as a solvent for α- cellulose. The phosphoric acid method, which is recent easy to control and less troublesome than other direct and also indirect method is used here.

Preparation of cellulose solution in phosphoric acid :

The entire cellulose sample is soluble in phosphoric acid at about 55± 2 ^{0}C. So the cellulose solution is prepared at a temperature of 55± 2 ^{0}C. In orders to prepare cellulose solution at a particular concentration of a particular cellulose sample, a calculated amount of both the cellulose and the phosphoric acids are taken in a 250 ml conical flasks. The flask is then shaken well for proper distribution of cellulose in phosphoric acid. When the cellulose swelled up, solution preparation is completed by heating the contents of the flask at 55± 2 ^{0}C in a water bath. When the solution becomes clear, it is suddenly cooled to 30 ^{0}C as soon as possible in a cooled water bath. The solution is then filtered and the filtrate is stored.

Apparatus Required:

1) The Ubbelohde suspended level viscometer. The viscometer was of such a dimension that the flow time of phosphoric acid (Used as a solvent for cel lulose) was 30 seconds.

2) Stop watch graduated in second and reading up to 0.05 second.

3) Electrically heated thermostatic water bath filled with stirrer. The temper ature of the bath was maintained at 55 ± 2 ^{0}C

Experimental technique:

Measurement of viscosity is carried out in an Ubbelohde suspended viscometer at 30 ^{0}C in a water bath. The bath temperature thermostatically controlled at 30 ± 0.2 ^{0}C. The viscometer is placed vertically in the water bath in such a way that the highest level of the liquid in the viscometer is at least 1 cm below the water level in the bath. Special precautions are taken to keep the viscometer free from grease and dust.

Before each measurement the viscometer containing experimental solution is allowed to stand for 5 minutes in the water bath to attain the temperature of the bath. The cellulose in phosphoric acid solutions of different concentrations is then prepared by dilution of the original solution. The concentration of which is determined previously. For each cellulose solutions the viscometric flow times are measured. The viscosity of phosphoric acid is determined at 25 ^{0}C solution. The relative viscosity of the α-cellulose in phosphoric acid solution is obtained by dividing the viscosity of the solution by viscosity of the solvent (phosphoric acid) at 25 ^{0}C

$$\eta_r = \frac{\eta_{solution}}{\eta_{solvent}}$$

Then the specific viscosity of the solution is $\eta_{sp} = \eta_r - 1$

Intrinsic viscosity[η] is then obtained by plotting η_{sp}/c against C and extra plotting to zero concentration, where C is the concentration in $\frac{g}{100ml}$ of the solution.

From the measured intrinsic viscosity the molecular weight of α-cellulose calculated by the equation.

$[\eta] = KM^a$ .....(12)

From the intercept of the plot logn against log [M], value of k and from the slope, value of "a" are found to be 1.78 X 10^{-5} and 1 respectively. The molecular weight of α-cellulose of *Agave Cantala* fibre can also be measured by indirect method.

Results and discussions

Isolation of the constituents of *Agave Cantala* (dakatia) fibre:

Table-1: Percentage of the composition of *Agave Cantala* fiber

Constituents	Amounts (%) dry basis
α-cellulose	70.00
Hemicellulose	16.60
Lignin	7.00
Fatty and waxy matters	2.00
Pectic matters	1.30
Aqueous extract	0.50
Miscellaneous	2.60
Total	100.00

From table-1 it is as evident that the composition of *Agave Cantala* fibre that the main constituents are α- cellulose (70%), Hemicellulose (16.6%) and Lignin 7% and the rest are very minor in proportion. So giving very little influence to the structure of *Agave Cantala* fibre, we know that lignin is the main constituents, which is responsible for yellowing of *Agave Cantala* fibre. Because *Agave Cantala fibre* contains high amount of α- cellulose and less amount of lignin. So it has strong background to carry out research for fastness properties of dyeing.

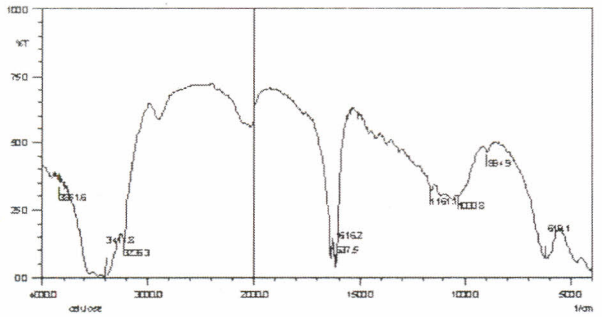

Fig - 1. The FTIR spectrum of α-cellulose

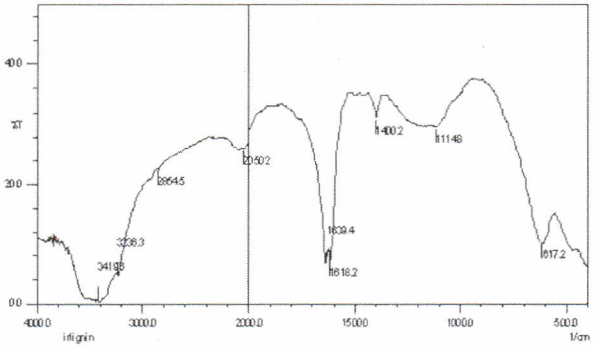

Fig - 2. The FTIR spectrum of Lignin.

The Infrared spectrum of α- cellulose and lignin by FTIR is given in the figure[1] and figure 2 represents the spectrum of α- cellulose and lignin which is extracted from *Agave Cantala* fibre. Lignin extracted from different sources have different structure. So, the spectrum peak can be changed, if it is extracted from different sources. From the analysis, spectrum of α-cellulose and lignin extracted from *Agave Cantala* fibre we have the following conclusions .

From the table below, it is evident that cellulose gives two main peak 3236.3 cm^{-1} for hydroxyl aldehydes and ketones. This gives the sufficient information for the functional group of cellulose. Lignin gives the same absorption of 3236.3 cm^{-1} for hydroxyl aldehydes and ketones but it gives another broad peak at 2854.5 cm^{-1}, which gives the evident that methoxy group present in lignin.

α- cellulose	Lignin
Wave number (cm-1)	Wave number (cm-1)
3411.8	3419.6
3236.3	3236.3
1637.5	2854.5
1616.2	2050.2
1161.1	1618.2
1033.8	1400.2
894.9	1114.8
619.1	617.2

Molecular weight determination of *Agave Cantala* fibre:
Cellulose solution in phosphoric acid were prepared with Agave Cantala fibre. Flow times of these cellulose solutions were measured at different concentrations. From the results the values of relative viscosityη_r, specific viscosityη_{sp} and reduced viscosity$\eta_{sp/c}$ were calculated. The intercept of straight line in plot of $\eta_{sp/c}$ v.s. concentration gave the intrinsic viscosity $[\eta]$ of cellulose. From the intrinsic viscosity we can calculate the molecular weight of *Agave Cantala* fibre.

Table-2 : Viscosity of cellulose solution in phosphoric acid at different concentration (for raw *Agave Cantala* fibre). (Open retting fibre)

Concentration C/gm/100ml.	Time of flow (second)	Relative viscosity $\frac{\eta}{\eta_o} = \frac{t}{t_o} = \eta_r$	Specific viscosity $\eta_{sp} = \eta_r - 1$	Reduced viscosity η_{sp}/c	Intrinsic viscosity $[\eta]$	Molecular Weight $(\overline{M}_n)$
Solvent H_3PO_4	188				2.4	134831
0.1	238.8	1.270	0.270	2.70		
0.2	297.3	1.581	0.581	2.90		
0.3	368.8	1.961	0.961	3.20		
0.4	458.7	2.439	1.439	3.50		
0.5	535	2.845	1.845	3.67		

Table-3: Viscosity of cellulose solution in phosphoric acid at different concentration (for bleached Agave Cantala fibre). (Open retting fibre)

Concentration C/gm/100ml.	Time of flow (second)	Relative viscosity $\frac{\eta}{\eta_o}=\frac{t}{t_o}=\eta_r$	Specific viscosity $\eta_{sp}=\eta_r-1$	Reduced viscosity η_{sp}/c	Intrinsic viscosity $[\eta]$	Molecular Weight $(\overline{M}_n)$
Solvent H_3PO_4	188					
0.1	235	1.25	0.25	2.50		
0.2	293.3	1.56	0.56	2.80		
0.3	357.2	1.90	0.90	3.00	2.25	126404
0.4	436.2	2.32	1.32	3.30		
0.5	526.4	2.80	1.80	3.60		

Table-4: Comparison of molecular weight of different fibres.

Name of fibre	Raw fibre ($\overline{M}n$)	Bleached fibre ($\overline{M}n$)
Agave Cantala fibre	134831	126404
Cotton tree fibre	119775	112752
Jute fibre	112359	106741
Mesta fibre	109550	102247
Banana fibre	143258	143258

Table:5. Comparison of composition of various fibre:

Constituents	Amounts percentage dry basis				
	Agave Cantala fibre	Cotton tree fibre	Jute fibre	Mesta fibre	Banana fibre
α- cellulose	70.00	66.50	61.50	58.60	65.8994
Hemicellulose	16.60	17.00	20.00	27.20	18.798
Lignin	7.00	7.50	13.20	11.00	7.5997
Fatty and waxy matters	2.00	2.50	1.60	0.50	2.602
Pectic matters	1.30	1.00	1.80	1.00	3.091
Aqueous extract	0.50	1.50	0.70	0.40	2.0099
Miscellaneous	2.60	4.00	1.20	1.30	-------

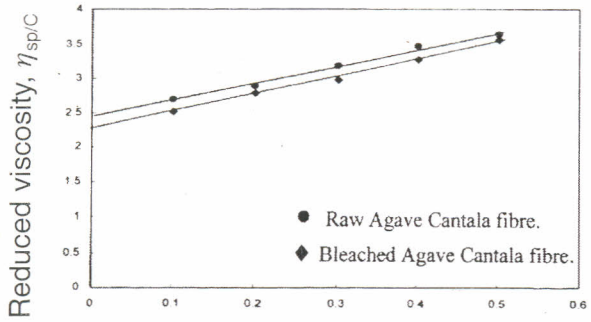

Fig-3: Plots of Concentration Vs. reduced viscosity for the intrinsic viscosity of raw and bleached Agave Cantala fibre.

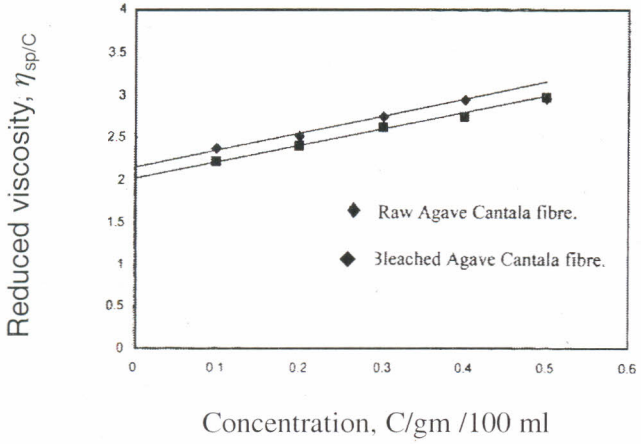

Fig-4: Plots of Concentration Vs. reduced viscosity for the intrinsic viscosity of raw and bleached Agave Cantala fibre (Boiled retting fibre).

Conclusions

From the isolation of *Agave Cantala* (Dakatia tree) fibre, it is seen that it contains high percentage of α-cellulose which is about 70%. The cell length of Agave Cantala fibre is higher. So, the absorption of dye molecules by the fibre is greater. The percentage of lignin in *Agave Cantala* fibre is 7 % which is less than jute fibre. So the yellowing occurs very slowly in *Agave Cantala* fibre which is desirable for fastness properties. Molecular weight of raw *Agave Cantala* fibre is 134831 and bleached fibre is 126404. The molecular weight of raw fibre is greater than that of bleached fibre.

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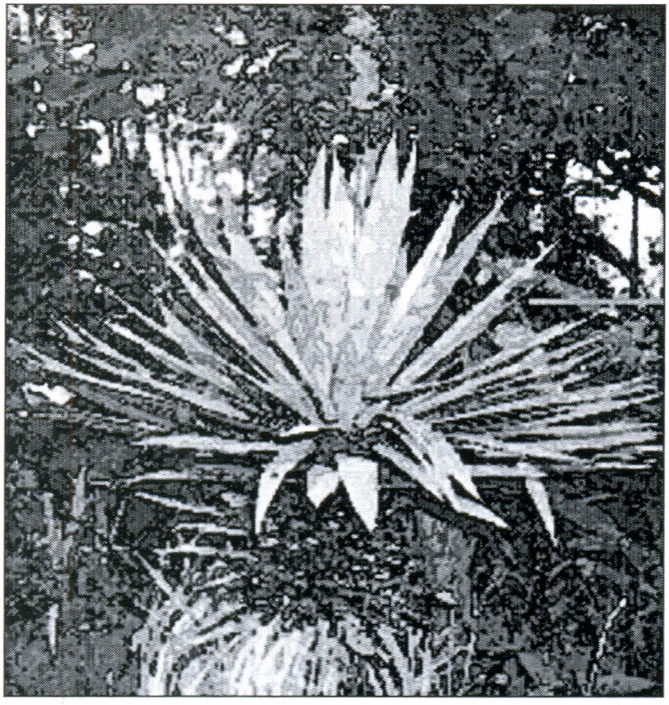

Agave Cantala (Dakatia) tree