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Extraction of Natural Dyes from Plants

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Abstract

The main idea of extracting dyes from plant (natural) sources is to avoid the environmental pollution. Present days with global concern over the use of eco-friendly and biodegradable materials, considerable research work is being undertaken around the world on the application of natural dyes in textile industry. The effluent problems of synthetic dyes occur not only during their application in the textile industry, but also during their manufacture and possibly during the synthesis of their intermediates and other raw materials. The use of natural dyes for textile dyeing purposes, decreased to a large extent after the discovery of synthetic dyes in1856. Dyes derived from natural sources have emerged as an important alternative to synthetic dyes. Analytical studies such as IR spectrophotometry were performed on the extract. The dyes produced were dyed on cotton fabric and tested for their color fastness to washing properties. Several mordants were also used for fixing the color on the fabrics. Moreover, the dyes obtained from the plant may also be alternative sources to synthetic dyes for the dyeing of natural cotton fiber. Therefore, this methodology is employed to first identify plants from which dyes can be extracted and to test the properties of the plant dyes and introduce them into the textile fabrics in a faster and effective manner.

Key words: Dyes, Textiles, Mordants, Biodegradable, Fabrics.

Introduction

Dyes are one of the most important uses of the plants. Recently, interest in the use of natural dyes has been growing rapidly due to the result of stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes. As a result with a distinct lowering in synthetic dyestuff costs, the natural dyes were virtually unused at the beginning of twentieth century (Kumaresan *et. al.*, 2011). Nowadays in most of the countries, natural dyeing is practiced only as a handcraft and synthetic dyes are being used in all commercial dyeing processes. However with the worldwide concern over the use of eco-friendly and biodegradable materials, the use of natural dyes has once again gained interest (Agarwal A, Goel A & Gupta K C, 1992).

Dyeing can be carried out in an alkaline bath, acidic bath or in a neutral bath. There are various reports available on different methods of mordanting on different fibers such as cellulosic, protenic and synthetic for dyeing with different natural dyes. Various kinds of shades like black to brown, green to yellow to orange, etc can be obtained by application of different mordants. Dyeing of cotton and silk with henna, indigo, marigold etc is reported. (Gulrajani *et al*, 1992). There is a growing interest in the revival of natural dyes in textile colouration (Mehanta z. & Osman et al, 2003 & 2004). In contrast, natural dyes are environmental friendly, exhibit better biodegradability and generally have a higher compatibility with the environment than synthetic dyes. (Ahlstrom *et al*, 2005). The process is economically viable as the raw materials are available at low cost and so cost of production is also very low. Similar findings were reported in Marigold, China rose and Bixa flower (Ibrahim et al, 1997).

Natural dyes/colorants derived from flora and fauna are believed to be safe because of its nontoxic, non-carcinogenic and biodegradable in nature (Cristea & Vilarem, 2003). Many tribes of Arunachal Pradesh have been using this plant species traditionally in combination with other plants for extraction and preparation of dyes utilizing indigenous processes (Mahanta & Tiwari, 2005). Natural dyes are now a days in demand not only in textile industry but in cosmetics, leather, food and pharmaceuticals. The rich biodiversity of our country has provided us plenty of raw materials, yet sustainable linkage must be developed between cultivation, collection and their use (Gokhale et al, 2004).

The application of natural dyes in textile industry are for various purposes, viz. dyeing of yarns, which are then woven into cloth, carpet or any other usable form; dyeing of cloths woven earlier; block printing, where the textile materials are printed with the help of printing blocks; Kalamkari where the "Kalam" or pen is used to draw beautiful designs on the cloth (Gopi, 2004). Although it is unlikely all dyestuffs will be produced solely from plants, it is an interesting and exciting prospect that one day a percentage of everyday colours could be naturally derived. The plant possesses many medicinal properties. Flowers are the most effective fermentation agent, used in Ayurvedic medicines (Kroes et al, 1990). Many investigations revealed that the use of combination of mordants in varying ratios gives different shades and different colour fastness results (Kumaresan, Palanisamy & Kumar, 2011). The flowers, which contain much of tannin, are Flame coloured and yield red/pink/brown/flame coloured shades of dye (depending upon the fabric used) in large amounts, therefore, utilized throughout India for dying silk and fabrics on a commercial scale. Light fastness of many natural dyes, particularly which are extracted from flower parts are found to be poor to medium (Samanta & Agarwal, 2009). India was a major exporter of herbal dyes but not so recently because of the ban on production of some of the synthetic dyes and intermediates in the developed countries due to pollution problem (Gaur, 2008).

There are primarily four sources from which natural dyes are available. *Specialized plant and animal sources* - Many plants and some animals have been identified as potentially rich in natural dye contents, and some of them have been used for natural dyeing. Normally natural dyes are extracted from the roots, stems, leaves, flowers, fruits of various plants, dried bodies of certain insects and minerals. The shade of the color a plant produces will vary according to time of the year the plant is picked, how it was grown, soil conditions, etc. *By-products (especially lac dye)* - The lac industry gives lac dye as a by-product, which is extracted from the effluent. *Chemical synthesis* - This involves synthesis of dyes with molecular structures identical to those of natural dyes.*Tissue or cell cultures by DNA transfer biotechnology* - Certain fungi such as Drechslera and Trichoderma produce anthraquinone derivatives as secondary metabolites (Salam M A, *et.al*., 2006).

Most of the natural dyes have no substantively on cellulose or other textile fibers without the use of a mordant. The majority of natural dyes need a mordanting chemical (preferably metal salt or suitably coordinating complex forming agents) to create an affinity between the fiber and dye or the pigment molecules of natural colorant. These metallic salts as mordant form metal complexes with the fibers and the dyes (Samanta *et. al.*, 2010). After mordanting, the metal salts anchoring to the fibers, attracts the dye/organic pigment molecules to be anchored to the fibers and finally creates the bridging link between the dye molecules and the fiber by forming coordinating complexes. Aluminum sulphate or other metallic mordants anchored to any fiber, chemically combine with certain mordantable functional groups present in the natural dyes and bound by coordinated/covalent bonds or hydrogen bonds and other interactional forces (Senthikumar *et. al*., 2002).

Materials and Methods

Materials Required

- Plant source
- Cotton cloth
- Bowls
- Beakers
- Conical flask
- Mordants
- Knife
- Vegetable grater
- Filter paper
- Tripod stand
- Mesh

Plants Used

- Peacock flower (*Caesalpinia pulcherima*)
- Bougainvillea (*Bougainvillea glabra*)
- Beetroot (*Beta vulgaris*)
- Red cabbage (*Brassica oleracea*)
- Onion skin (*Allium cepa*)

Extraction of Dyes from the Plants

The workstation was covered with the newspaper. Fresh vegetables and flowers especially with bright colors were selected. The skin was peeled and grated using vegetable grater for better extraction. 5-10 gms of grated vegetables was added to 100ml of distilled water and was boiled well until the dye was released in water. This can be done for

at least 15-20 mins for the complete extraction. Flowers can be cut into small pieces and step 4 & 5 can be followed. After the dye is extracted it is stored in the refrigerator for further use.

Preparation of Mordant

Alum

0.748g of Alum and 0.187g of Washing soda were mixed in 100ml of water and was stored for further use.

Vinegar

50 ml of 5% ascetic acid is mixed with 100ml of water. From that 25ml of it were taken and mixed with 100ml of distilled water.

Salt

5 g of Sodium chloride was mixed in 100ml of distilled water and was used as a mordant and stored for further use.

The vegetable is prepared. In another pot, the "fixative" recipe is prepared. It is brought to a light boil and the fabric is added. The fabric is simmed in the fixative for at least an hour. After an hour, the fabric is carefully pulled out from the simmering fixative and is wrung out completely. Next, a new pot with the "fixed" fabric is taken. The fixed fabric is immersed in the dye which is already extracted. It is then boiled and simmed until the fabric takes up the dye at least for an hour. The fabric is then removed and placed on the newspaper or tile to dry. Since the fabric takes up the dye it is ready to be used for commercial purpose.

Phytochemical Test

A small amount of the substance is taken and a few drops of alcohol and few drops of ferric chloride were added. It is shaken well till the appearance of **greenish yellow** color which indicates the presence of **phenol.** 0.5g of substance is mixed with 20ml of distilled water and it is boiled for some time and then few drops of 0.1% ferric chloride was added. It is mixed well till the appearance of **brownish green colour** which will indicate the presence of **tannin**. A small amount of the substance is mixed with sudan III which results in **shining orange** which indicates the presence of **fat and fixed oil**. Small amount of substance is taken and mixed with 10% sodium hydroxide which results in **greenish brown colour** which indicates the presence of **flavonoids.** This compound is present in almost all the brightly colored flowers, fruits, and vegetables. A small amount of the substance is shaken with a small amount of distilled water. Appearance of **frothing** will indicate the presence of **saponin**. Small amount of substance is mixed well with a few drops of chloroform. Then, a drop of acetic acid id added and the mixture is heated for few minutes after which few drops of concentrated sulphuric acid is added. Appearance of **orange** color will indicates the presence of **steroids**. The substance is mixed with few drops of concentrated hydrochloric acid; appearance of **green** color indicates the presence of **quinine.** Small amount of substance is mixed with iodine crystals and then a few drops of conc. sulphuric acid is added. Appearance of **brown color** indicates the presence of **cellulose.** The substance is mixed with 2ml of chloroform and then few drops of concentrated sulphuric acid is added. **Light orange color** appearance confirms the presence of **terpenoids.** The substance is mixed with 2ml of glacial acetic acid and one drop of ferric chloride is added followed by 1ml of conc. sulphuric acid and is mixed well. Appearance of **brown color** indicates the presence of **glycosides (Table 1).**

Table.1 Phytochemical Test

Phenol	-	-	+	+	+	-
Tannin	-	-	-	-	-	-
Flavonoids	+	+	+	+	+	+
Saponin	-	-	-	-	-	-
Steroids	+	-	-	-	-	-
Quinone	-	-	-	-	-	-
Cellulose	+	+	+	+	+	+
Terpenoids	+	-	-	-	-	-
Glycosides	+	-	-	-	-	-
Fat and fixed oil	-	-	-	-	-	-

Results and Discussion

The dye extracted from the plant and the color of the dye is depended on the compounds present in the respective plant. The dye is then used in the cotton fabric for the fixation of color. The cloth which is displayed above got fixed to the respective dye with the help of mordant. Different mordant were used for different plants. The mordant used were

Peacock flower	–	Vinegar
Beetroot	–	Salt
Onion skin	–	Alum
Red cabbage	–	Vinegar
Bougainvillea	–	Alum and Cream of Tartar
Papaya leaf	–	Salt

These mordants when added to the dye gave different shades of color and thus enable us to make different types of shades from one plant using the mordant. The mordants alum and cream of tartar is directly added to the dye whereas, in case of vinegar and salt the cloth is treated with those mordants and then it is immersed into the dye. This gives better results than directly adding the mordant into the dye. Because, when treated using heat the mordant sticks into the fabric well again and when the cloth is treated with the dye, the color does not fade and stays forever (**Figures 2 – 10**).

Figure 2 : Caesalpinia flower

Figure 3 : Beetroot

Figure 4 : Onion Skin

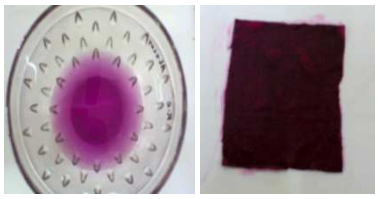

Figure 5 : Red Cabbage

Figure 6 : Bougainvillea

Figure 7 : Papaya Leaf

Figure.8: Positive result for Caesalpinia

Figure.9: Positive result for Beetroot

Figure.10: Positive result for Red cabbage

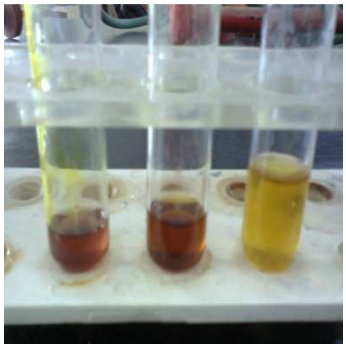

Figure.11: Positive result for Bougainvillea

Principles of Infrared (IR) Absorption

Infrared spectroscopy is the measurement of the wavelength and intensity of the absorption of infrared light by a sample. The infrared spectra usually have sharp features that are characteristic of specific types of molecular vibrations, making the spectra useful for sample identification (Figures 14 – 17).

Characteristic IR Bands

X-H Vibrations	Bond	Wave numbers (cm^{-1})
Hydroxyl	O-H	3610-3640
Aromatic rings	C-H	3000-3100
Amines	N-H	3300-3500
Alkenes	C-H	3020-3080
Alkanes	C-H	2850-2960
Triple bonds	C□C	2500-1900
Double bonds	C=C	1900-1500
Deformation/heavy atoms		1500-

Quantitative Analysis of Plants

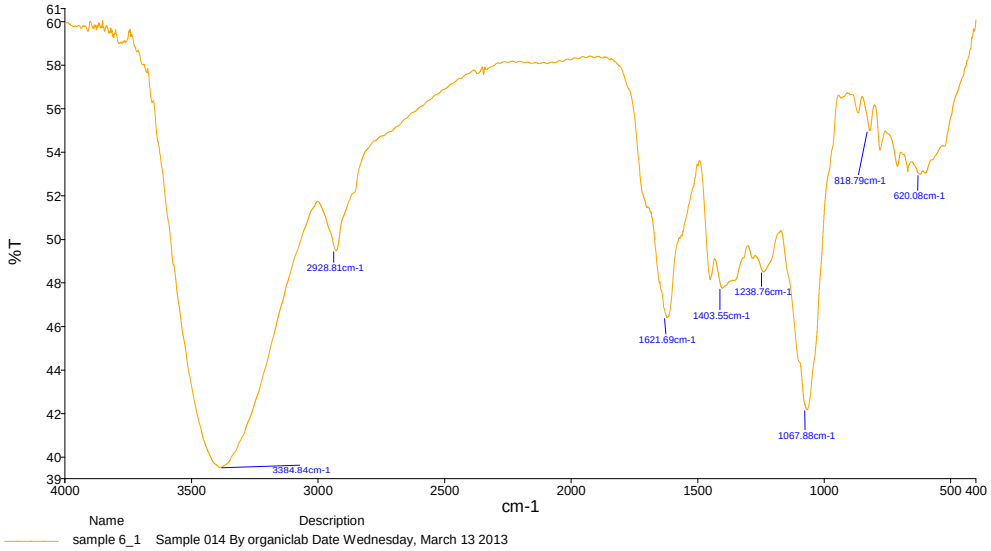

Figure.12: IR Spectroscopy of Peacock flower (caesalpinia)

Interpretation

The IR spectroscopy of Peacock Flower shows the range of
Phenol compound - 3384nm (3200-3550nm)
Alkanes - 2928nm (2850-3000nm)

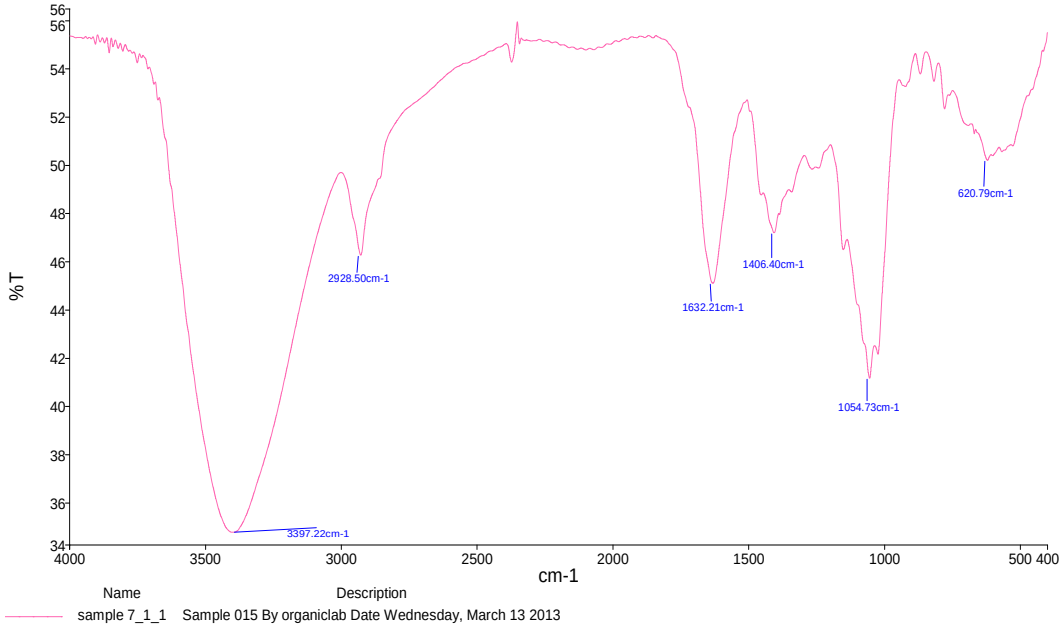

Figure.13: IR Spectroscopy of Beet Root

Interpretation

The IR spectroscopy of Beet Root shows the range of
Phenol compound - 3397nm (3200-3550nm)
Alkanes - 2928nm (2850-3000nm)
Alkenes - 1632nm (1630-1680nm).

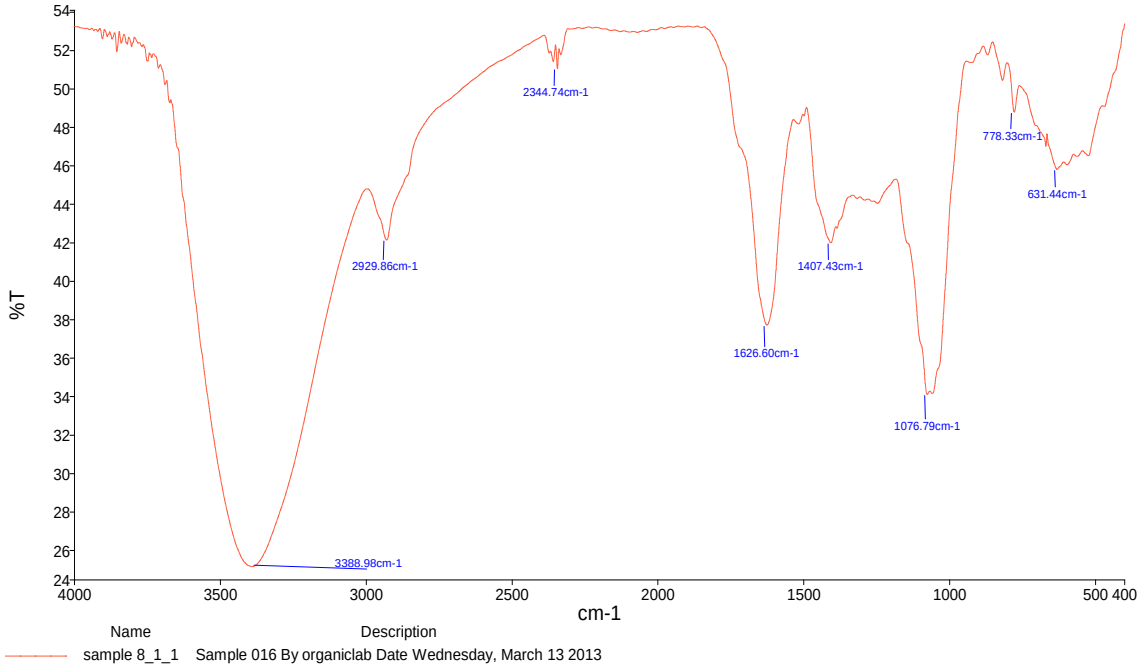

Figure.14: IR Spectroscopy of Onion Skin

Interpretation

The IR spectroscopy of Onion Skin shows the range of
Phenol compound - 3388nm (3200-3550nm),
Alkanes - 2929nm (2850-3000nm), and
Amines - 1076nm (1000-1250nm).

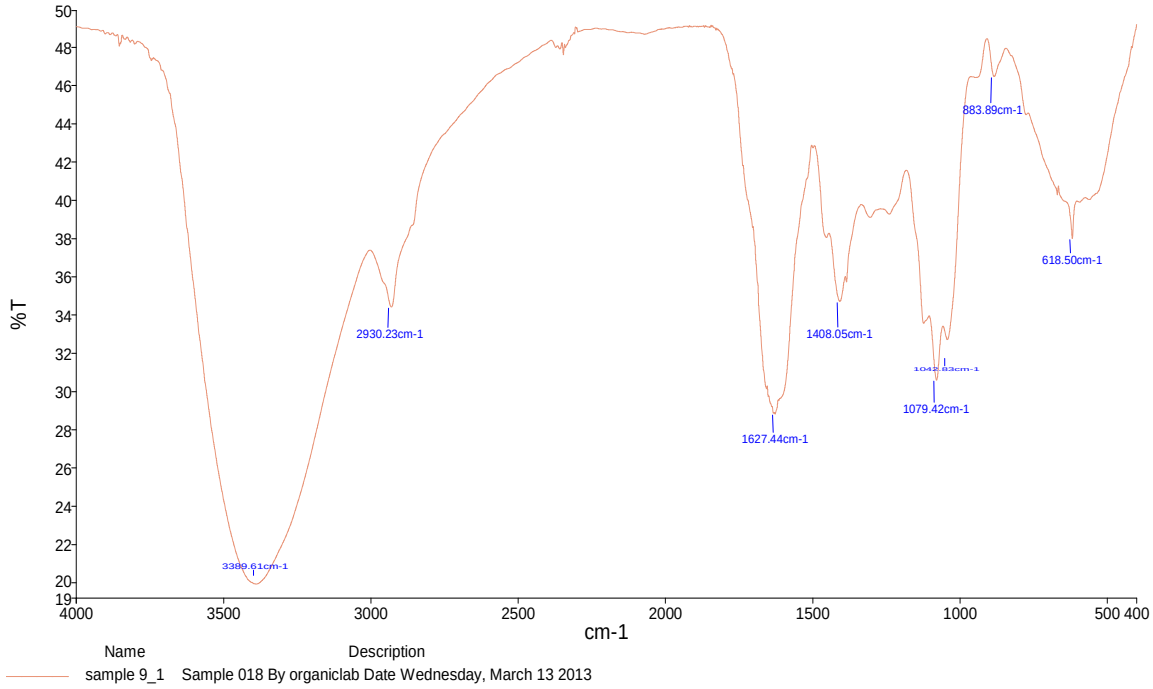

Figure.15: IR Spectroscopy of Red Cabbage

Interpretation

The IR spectroscopy of Red cabbage shows the range of
Phenol compound - 3389nm (3200-3550nm),
Alkanes - 2930nm (2850-3000nm).

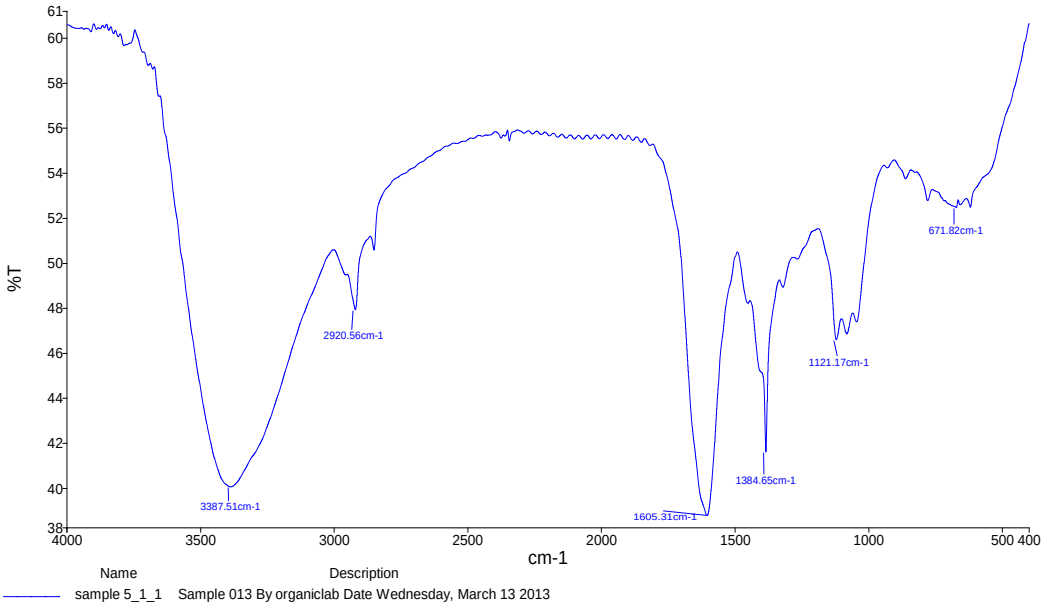

Figure.16: IR spectroscopy of Papaya leaf

Interpretation

The IR spectroscopy of Papaya Leaf shows the range of
Phenols compound - 3387nm (3200-3550nm)
Alkanes - 2920nm (2850-3000nm)
Amines - 1121nm (1000-1250nm).

Summary

Different parts of the plants were used for the extraction of dyes such as leaves, flowers, vegetables, etc and different types of mordants were used for fixing the dye into the fabric. In ancient days people have used natural dyes to paint their caves. Over 15,000 BC man began to produce those natural dyes which have been used in textiles as well. In order to understand the art and history of dyeing, we must first understand the process of dyeing itself. Natural dyes can be broken down into two categories: substantive and adjective. Most ancient and medieval dyers mordanted their yarns and fabrics before dyeing them. Different fibers also have different tendencies to absorb natural and synthetic dyes. Wool, a protein-based fiber, has been found in Europe dating back to 2000 BC. In Europe the art of dyeing rose to new heights with the diversity of climate, culture and migration or invasion waves. Eventually, the old natural dyes lost popularity in favor of the newer synthetic ones.

The alchemy of colors started from early time. With the modern phases of development, dyes have become the most important resources, owing to their multifarious utilization, including an emerging branch of medicine i.e., Chromo therapy which greatly depends on natural coloring dyes. Usually, methods of collection and extraction of dyes are still crude and traditional with only a few experts related to cottage industries being well versed with dyeing procedures. Indigenous traditional knowledge on various resources including dye yielding plants is very essential for rural based development and future bioprospecting, provided proper precautionary measures are considered for sustainability, conservation and value based selection of use pattern. Nowadays most of the natural dyers are interested to use natural dye materials in the same ways used for synthetic dyes. Textile dyers must know the chemistry of these natural colors and its added advantages of medicinal values. Use of suitable binary or ternary mixtures of similar or compatible natural dyes for coloring natural eco-friendly textiles in variety of soothing / uncommon shades with eco-friendly mordants and finishing agents are the most desirable product of the customers for future. So, a textile dyer must know the effects of variability for extraction, mordanting and dyeing and should follow only the standardized recipe for selection fiber mordant natural dye system to get reproducible color yield and color matching besides to follow different eco-friendly ways to improve color fastness to a possible extent. Thus with the worldwide concern over the use of eco-friendly and biodegradable materials, the use of natural dyes has undoubtedly once again gained interest and momentum.

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