AMERICAN JOURNAL OF PHARMTECH RESEARCH

Journal home page: http://www.ajptr.com/

Isolation of Natural Dyes from the Flower of *Hibiscus Rosa-Sinensis*

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ABSTRACT

Natural dyes are now-a-days very common for every cloth industry because the harmfulness of synthetic dyes on the skin of human beings. In cotton, silk and also in wool these colours are stable and very much eco-friendly to human skin. So now, several attempts are taken by the scientists through out the world to isolate natural dyes from different vegetables and flowers. Here in this study an attempt has been taken to isolate natural dyes from the flowers of *Hibiscus rosa-sinensis*. In the result, three different colours like blue, purple and green were prepared from the above said flowers and they are well stable on cotton cloths after washing by hot water and soap too. The intensity of the three colours are also high, they are bright and really eco-friendly to the human skin.

Keywords: Natural dyes, *Hibiscus rosa-sinensis*, mordant

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Received 11 April 2012, Accepted 26 April 2012

Please cite this article in press as: Bose S *et al.*, Isolation of Natural Dyes from the Flower of *Hibiscus Rosa-Sinensis*. American Journal of PharmTech Research 2012.

INTRODUCTION

Natural dyes are dyes or colorants derived from plants, invertebrates, or minerals. The majority of natural dyes are vegetable dyes from plant sources – roots, berries, bark, leaves, and wood — and other organic sources such as fungi and lichens. Archaeologists have found evidence of textile dyeing dating back to the Neolithic period. In China, dyeing with plants, barks and insects has been traced back more than 5,000 years. The essential process of dyeing changed little over time. Typically, the dye material is put in a pot of water and then the textiles to be dyed are added to the pot, which is heated and stirred until the color is transferred. Textile fiber may be dyed before spinning (dyed in the wool), but most textiles are yarn-dyed or piece-dyed after weaving. Many natural dyes require the use of chemicals called mordants to bind the dye to the textile fibers; tannin from oak galls, salt, natural alum, vinegar, and ammonia from stale urine were used by early dyers. Many mordants, and some dyes themselves, produce strong odors, and large-scale dye works were often isolated in their own districts[1].

Throughout history, people have dyed their textiles using common, locally available materials, but scarce dyestuffs that produced brilliant and permanent colors such as the natural invertebrate dyes Tyrian purple and crimson kermes became highly prized luxury items in the ancient and medieval world. Plant-based dyes such as woad, indigo, saffron, and madder were raised commercially and were important trade goods in the economies of Asia and Europe. Across Asia and Africa, patterned fabrics were produced using resist dyeing techniques to control the absorption of color in piece-dyed cloth. Dyes from the New World such as cochineal and logwood were brought to Europe by the Spanish treasure fleets, and the dyestuffs of Europe were carried by colonists to America.

The discovery of man-made synthetic dyes in the mid-19th century triggered the end of the large-scale market for natural dyes. Synthetic dyes, which could be produced in large quantities, quickly superseded natural dyes for the commercial textile production enabled by the industrial revolution, and unlike natural dyes, were suitable for the synthetic fibers that followed. Artists of the Arts and Crafts Movement preferred the pure shades and subtle variability of natural dyes, which mellow with age but preserve their true colors, unlike early synthetic dyes, and helped ensure that the old European techniques for dyeing and printing with natural dyestuffs were preserved for use by home and craft dyers. Natural dyeing techniques are also preserved by artisans in traditional cultures around the world.

A variety of plants produce red dyes, including a number of lichens, henna, alkanet or dyer's bugloss (*Alkanna tinctoria*), asafoetida and madder. Madder (*Rubia tinctorum*) and related plants of the Rubia family are native to many temperate zones around the world, and have been used as a source of good red dye (rose madder) since prehistory. Madder has been identified on linen in the tomb of Tutankhamun, and Pliny the Elder records madder growing near Rome. Madder was a dye of commercial importance in Europe, being cultivated in Holland and France to dye the red coats of military uniforms until the market collapsed following the development of synthetic alizarin dye in 1869. Madder was also used to dye the "hunting pinks" of Great Britain.

Turkey red was a strong, very fast red dye for cotton obtained from madder root via a complicated process involving "sumac and oak galls, calf's blood, sheep's dung, oil, soda, alum, and a solution of tin." Turkey red was developed in India and spread to Turkey. Greek workers familiar with the methods of its production were brought to France in 1747, and Dutch and English spies soon discovered the secret. A sanitized version of Turkey red was being produced in Manchester by 1784, and roller-printed dress cottons with a Turkey red ground were fashionable in England by the 1820s.

Munjeet or Indian madder (*Rubia cordifolia*) is native to the Himalayas and other mountains of Asia and Japan. Munjeet was an important dye for the Asian cotton industry and is still used by craft dyers in Nepal[2].

Puccoon or bloodroot (*Sanguinaria canadensis*) is a popular red dye among Southeastern Native American basketweavers. Choctaw basketweavers additionally use sumac for red dye.Coushattas artists from Texas and Louisiana used the water oak (*Quercus nigra L.*) to produce red. A delicate rose color in Navajo rugs comes from fermented prickly pear cactus fruit, *Opuntia polycantha*. Navajo weavers also use rainwater and red dirt to create salmon-pink dyes. Dyes that create reds and yellows can also yield oranges. Navajo dyers create orange dyes from one-seeded juniper, *Juniperus monosperma*, Navajo tea, *Thelesperma gracile*, or alder bark. Yellow dyes are "about as numerous as red ones", and can be extracted from saffron, pomegranate rind, turmeric, safflower, onion skins, and a number of weedy flowering plants. There is limited evidence of the use of weld (Reseda luteola), also called mignonette or dyer's rocket before the Iron Age, but it was an important dye of the ancient Mediterranean and Europe, and is indigenous to England. Two brilliant yellow dyes of commercial importance in Europe from the 18th century are derived from trees of the Americas: quercitron from the inner bark of oaks native to North America and fustic from the dyer's mulberry tree (*Maclura tinctoria*) of the West Indies and Mexico.

In rivercane basketweaving among Southeastern tribes, butternut (*Juglans cinerea*) and yellow root (*Xanthorhiza simplicissima*) provide a rich yellow color. Chitimacha basket weavers have a complex formula for yellow that employs a dock plant (most likely Rumex crispus) for yellow. Navajo artists create yellow dyes from small snake-weed and brown onion skins, and rubber plant (*Parthenium inca*num). Rabbitbush (Chrysothamnus) and rose hips produce pale, yellow-cream colored dyes.

If plants that yield yellow dyes are common, plants that yield green dyes are rare. Both woad and indigo have been used since ancient times in combination with yellow dyes to produce shades of green. Medieval and Early Modern England was especially known for its green dyes. The dyers of Lincoln, a great cloth town in the high Middle Ages, produced the Lincoln green cloth associated with Robin Hood by dyeing wool with woad and then over dyeing it yellow with weld or dyer's greenweed (*Genista tinctoria*), also known as dyer's broom. Woolen cloth mordanted with alum and dyed yellow with dyer's greenweed was overdyed with woad and, later, indigo, to produce the once-famous Kendal green. This in turn fell out of fashion in the 18th century in favor of the brighter Saxon green, dyed with indigo and fustic.

Soft olive greens are also achieved when textiles dyed yellow are treated with an iron mordant. The dull green cloth common to the Iron Age Halstatt culture shows traces of iron, and was possibly colored by boiling yellow-dyed cloth in an iron pot.Indigenous peoples of the Northwest Plateau in North America used lichen to dye corn husk bags a beautiful sea green. Navajo textile artist Nonabah Gorman Bryan developed a two-step process for creating green dye. First the Churro wool yarn is dyed yellow with sagebrush, Artemisia tridentata, and then it is soaked in black dye afterbath. Red onion skins are also used by Navajo dyers to produce green.

Blue colorants around the world were derived from indigo dye-bearing plants, primarily those in the genus Indigofera, which are native to the tropics. The primary commercial indigo species in Asia was true indigo (*Indigofera tinctoria*). India is believed to be the oldest center of indigo dyeing in the Old World. It was a primary supplier of indigo dye to Europe as early as the Greco-Roman era. The association of India with indigo is reflected in the Greek word for the dye, which was indikon. The Romans used the term indicum, which passed into Italian dialect and eventually into English as the word indigo. In Central and South America, the important blue dyes were Añil (*Indigofera suffruticosa*) and Natal indigo (*Indigofera arrecta*). In temperate climates including Europe, indigo was obtained primarily from woad (*Isatis tinctoria*), an indigenous plant of Assyria and the Levant which has been grown in Northern Europe over 2,000 years, although from the 18th century it was mostly replaced by superior Indian indigo imported by the British East India Company. Woad was carried to New England in the 17th century and used extensively in America until native stands of indigo were discovered in Florida and the Carolinas. In Sumatra, indigo dye is extracted from some species of Marsdenia. Other indigo-bearing dye plants include dyer's knotweed (*Polygonum tinctorum*) from Japan and the coasts of China, and the West African shrub *Lonchocarpus cyanescens*.

In medieval Europe, purple, violet, murrey and similar colors were produced by dyeing wool with woad or indigo in the fleece and then piece-dyeing the woven cloth with red dyes, either the common madder or the luxury dyes kermes and cochineal. Madder could also produce purples when used with alum. Brazilwood also gave purple shades with vitriol (sulfuric acid) or potash. Choctaw artists traditionally used maple (Acer sp.) to create lavender and purple dyes. Purples can also be derived from lichens, and from the berries of White Bryony from the northern Rocky Mountain states and mulberry (*Morus nigra*) (with an acid mordant). Cutch is an ancient brown dye from the wood of acacia trees, particularly Acacia catechu, used in India for dyeing cotton. Cutch gives gray-browns with an iron mordant and olive-browns with copper. Black walnut (*Juglans nigra*) is used by Cherokee artists to produce a deep brown approaching black. Today black walnut is primarily used to dye baskets but has been used in the past for fabrics and deerhide. Juniper, Juniperus monosperma, ashes provide brown and yellow dyes for Navajo people,as do the hulls of wild walnuts (*Juglans major*). Choctaw dyers use maple (Acer sp.) for a grey dye. Navajo weavers create black from mineral yellow ochre mixed with pitch from the piñon tree (*Pinus edulis*) and the three-leaved sumac (*Rhus trilobata*).They also produce a cool grey dye with blue flower lupine and a warm grey from Juniper mistletoe (*Phoradendron juniperinum*). The dye lake is an insoluble molecule formed when the complex of dye and mordant are combined, which then attaches to the substrate. Mordants increase the fastness of the dye since the larger molecule is now bonded to the fiber[3].

The term "lake" is derived from the term lac, the secretions of the Indian wood insect *Laccifer lacca* (formerly known as the *Coccus lacca*. This is the same insect from which shellac is obtained. The type of mordant used can change the colour of both the dye-plus-mordant solution and influence the shade of the final product.

Unlike cotton, wool is highly receptive toward mordants. Due to its amphoteric nature wool can absorb acids and bases equally effectively. When wool is treated with a metallic salt it hydrolyses the salt into an acidic and basic component. The basic component is absorbed at –COOH group and the acidic component is removed during washing. Wool also has a tendency to absorb fine precipitates from solutions; these cling to the surface of fibres and dye particles attached to these contaminants result in poor rubbing fastness. Like wool, silk is also amphoteric and can absorb both acids as well as bases. However, wool has thio groups (-SH) from the cystine amino acid, which act as reducing agent and can reduce hexavalent chromium of potassium dichromate to trivalent form. The trivalent chromium forms the complex with the fibre and dye. Therefore potassium dichromate cannot be used as mordant effectively.

Hibiscus rosa-sinensis, known colloquially as the Chinese hibiscus, China rose and shoe flower, is an evergreen flowering shrub native to East Asia.It is widely grown as an ornamental plant throughout the tropics and subtropics. The flowers are large, generally red in the original varieties, and firm, but generally lack any scent.

Pink-Red *Hibiscus rosa-sinensis* are common Hibiscus flower in India.(Figure 1) Hibiscus flower preparations are used for hair care. The flowers themselves are edible and are used in salads in the Pacific Islands. The flowers are used to shine shoes in parts of India. It is also a pH indicator. China rose indicator turns acidic solutions to magenta/dark pink and basic solutions to green. It is also used for the worship of Devi and especially the red variety takes an important part in tantra. In Indonesia, these flowers are called "kembang sepatu", which literally means "shoe flower". *Hibiscus rosa-sinensis* is considered to have a number of medical uses in Chinese herbology[4]. Here in this study an attempt has been taken to isolate natural dyes(Figure 2) from the flower of *Hibiscus rosa-sinensis* and its application on the cotton and silk.

Figure 1: *Hibiscus rosa-sinensis*

Figure 2: Natural dyes

MATERIALS AND METHODS

Extraction of natural dye from the flower of *Hibiscus rosa-sinensis* were carried out through boiling and solvent extraction process.

The flower of *Hibiscus rosa-sinensis* were collected from the Medicinal Plants' Garden, Gupta College of Technological Sciences, Asansol.

Boiling extraction[5-11]

In the case of boiling method, the materials were cut into small pieces, soaked in distilled water and heated for 1hour at 100˚C. The solution was then filtered and mixed with 2% of selected mordant based on weight fabric (o.w.f.). The dye liquor was used to dye silk fabric at 80˚C for 1 hour. Soap at boil was carried out to remove unattached dyes for 5 minutes.

Solvent extraction[5-11]

In the case of solvent extraction method, the materials were cut into small pieces and soaked in methanol and placed in dark room for two days at room temperature. The solution was then filtered and mixed with 2% of selected mordant based on weight fabric (o.w.f.). The dye liquor was used to dye silk fabric at 80^{o}C for 1 hour. Soap at boil was carried out to remove unattached dyes for 5 minutes.

Colour fastness to washing[5-11]

Colour fastness to washing is the ability to retain its colour after washing. The rating of wash fastness is from 1 to 5 where rating of 1 is the worst and 5 is the best.

RESULTS AND DISCUSSION

The different results of isolation of natural dyes from *Hibiscus rosa-sinensis* and their stability and colour's intensity are described in Table 1, 2 and 3. The present experiment has successfully produced three colours out of one source through boiling and solvent extractions. In Figure. 3, it has been shown that three different colours was produced from the flower of *Hibiscus rosa-sinensis* and in Figure 4, the histogram of colour intensity has been shown where it is clear that the intensity of blue colour is higher than the intensity of purple and green. The study revealed the production of varied natural colours from plant pigments. The present experiment emphasizes the utilization of waste flowers for value-addition. Future investigation will be directed towards developing eco-friendly dyes for textile and handloom industries. This will also have an impact on the economic growth of the rural weaver communities. The finding will benefit dyeing and printing industry such as batik manufacturers and can also benefit local fashion industry in promoting the use of natural colourant. Moreover, the dyes obtained from the plants may also be alternative sources to synthetic dyes for the dyeing of natural silk and cotton fiber. The natural dyes are safe because of its non-toxic, non-carcinogenic and biodegradable in nature.

Table 1: Result for the method of Boiling Extraction

Mordant	Piece of cloth	Colour produce
Alum	Cotton	Purple colour
	Silk	No colour
Wood ash	Cotton	No colour
	Silk	No colour

Table 2: Result for the method of Solvent Extraction

Mordant	Piece of cloth	Colour produce
Alum	Cotton	Green colour
	Silk	No colour
Wood ash	Cotton	Reddish blue colour
	Silk	No colour

Table 3: Colour fastness to washing Test on dyed cotton and silk fabrics

Method of Staining	Solvent Used	Mordant Used	Piece of Cloth	Colour Produce	Colour fastness washing test
Boiling Extraction Method	Distilled Water	Alum	Cotton	Purple	5
			Silk	-	-
		Wood Ash	Cotton	-	-
			Silk	-	-
Solvent Extraction Method	Methanol	Alum	Cotton	Green	5
			Silk	-	-
		Wood Ash	Cotton	Reddish Blue	5
			Silk	-	-

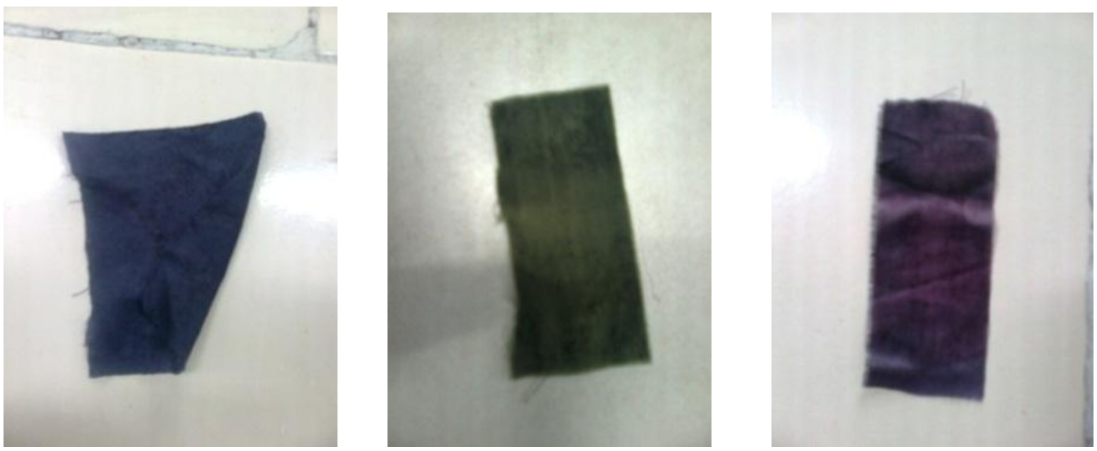

Figure 3: Blue, green and purple colours natural dyes produced from the flower of *Hibiscus rosa-sinensis*

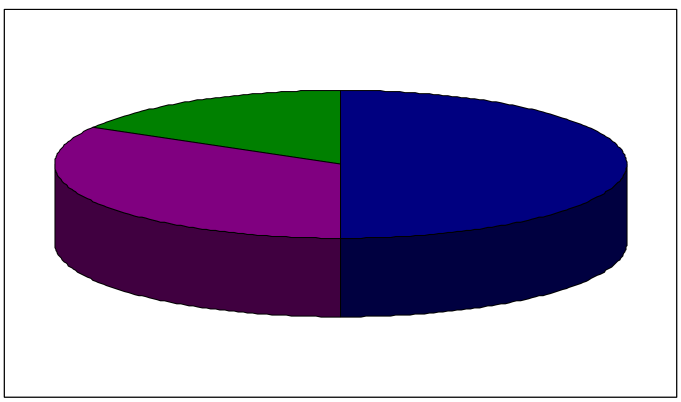

Figure. 4: Histogram of colour intensity of natural dyes produced from *Hibiscus rosa-sinensis*

CONCLUSION

From the result, three different colours like blue, purple and green were prepared from the above said flowers and they are well stable on cotton cloths after washing by hot water and soap too. The intensity of the three colours are also high, they are bright and really eco-friendly to the human skin.

ACKNOWLEDGEMENT

Authors are really thankful to the honorable Principal of Gupta College of Technological Sciences, Asansol, Dr. Kalyan Kumar Sen and honorable Chairman of Trinity Trust, Prof. Debesh Chandra Majumdar, for offering the proper environment, laboratory instruments and reagents, internet facilities and overall administration which are very much suitable for this project work. Authors would also render their thanks to all faculty members of this august institution for their help to complete this project work.

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