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## Effective natural dye extraction from different plant materials using ultrasound

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#### 1. Introduction

#### 1.1. Dyes and coloring materials

Highly colored substances, widely known as colorants, can be used to impart color to an infinite variety of materials described technically as substrates. Colorants can be subdivided into dyes and dyeing is a common application used for coloring fibrous substances and termed as staining in the case of biological materials. The dye is generally applied in an aqueous solution, and may require a mordant to improve the fastness of the dye on the fibre.

Synthetic dyes quickly replaced the traditional natural dyes and being used widely in textile, food and leather dyeing. Azo dyes are synthetic organic colorants, characterized by chromophoric azo groups (-N=N-) prepared by generally known unit process of Diazotization and coupling reaction. However, whereas azo dyes are relatively resistant to degradation under aerobic conditions, they can be readily reduced to form aromatic amines under anaerobic conditions. Various health problems associated with azo dyes are well reported (Ahlström et al., 2005; Osman et al., 2004; Chatterjee et al., 2007; Alves et al., 2007). In the case of reactive dyes, as much as 50% of the initial dye load is present in the dye bath effluent (Rai et al., 2005). In order to overcome these problems, bio remediation for azo dyes as end-of-pipe treatment method has been reported (Oztürk and Abdullah, 2006; Fu and Viraraghavan, 2001; Husain,

#### ABSTRACT

Dyes derived from natural sources have emerged as an important alternative to synthetic dyes. Therefore, there is a need for developing better solid–liquid extraction techniques for leaching natural colorants from plant materials for applications in plant research, food as well as dyeing industries. The influence of ultrasound on natural colorant extraction from different potential dye yielding plant materials has been studied in comparison with magnetic stirring process as control. The color yielding plant materials used in the present study include Green wattle bark, Marigold flowers, Pomegranate rinds, 4'o clock plant flowers and Cocks Comb flowers. Analytical studies such as UV–VIS spectrophotometry and gravimetric analysis were performed on the extract. The results indicate there is a significant 13–100% improvement in the extraction efficiency of the colorant obtained from different plant materials due to the use of ultrasound. Therefore, this methodology could be employed for extracting coloring materials from plant materials in a faster and effective manner.

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2006). The effluent treatment can also be done using suitable adsorbents (Crini, 2006), but create disposal problem. Therefore, there is no currently available effective method for removal of these toxic dye effluent wastes. These factors tend to restrict the use of these methods in routine monitoring of industrial effluent discharges to the amines, which have been proven as health hazards, namely the carcinogens (Pinheiroa et al., 2004). With environmental concerns rising on the utilization of synthetic chemical dyes natural dyes offers scope for eco-friendly way for food coloration and dyeing of fibrous materials such as textiles or leather and hence can be also used in specialty applications where non-toxicity is a must.

#### 1.2. Natural dye

Although synthetic dyeing methods have taken over in the last century, dyeing materials are still abounding in the natural world today. The global demand for natural dyes world over is about 10,000 tonnes, which is equivalent to 1% of the world synthetic dyes consumption. This is expected to rapidly grow in near future. Natural dyes can be derived from almost anything—plants, minerals, and even some insects. Most natural dye colors are found in the roots, bark, leaves, flowers, skins, and shells of plants. The advantage of natural dyes is eco-friendly, i.e., they do not create any environmental problems at the stage of production or use and maintains ecological balance. The recent ban on the use of azo dyes by European Union has also increased the scope for the use of natural dyes.

Studies regarding availability of natural dye yielding plants in north east region of India and various indigenous extraction proce-

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dures followed by the local communities were reported (Mehanta and Tiwari, 2005; Bhuyan and Saikia, 2004). These natural dyes can be used for coloring food, cosmetics, and clothing for children. Unlike the synthetic dyes, which are carcinogenic, these dyes are very eco-friendly and hence can be used in specialty applications where non-toxicity is a must.

#### 1.3. Natural dye extraction

Extraction of coloring matter is a solid-liquid leaching process involving mass transfer problem. Since the coloring matter is tightly bound with plant cell membranes, extraction could be better by way of some improved methods such as ultrasound. There is a need for novel techniques to improve the major mechanism of natural dye extraction such as rupture the cell wall, release of natural dye and improve the transport of dye in to the external medium. There is also a need for maximizing the efficiency of natural colorant extraction and its application in order to conserve the natural resources available. The gamma ray irradiation technique studied to improve the extraction efficiency led to possible degradation and instability of coloring matter (Nayak et al., 2006). The use of electric pulse studied for the same (Fincan et al., 2004) may involve operational difficulties. Therefore, the use of power ultrasound to improve the natural dye extraction from various plant materials has been reported in this paper. Details regarding different natural dye yielding plants under present investigation are given in Table 1.

#### 1.3.1. Use of power ultrasound

Ultrasound is classified according to frequency range as power ultrasound (20–100 kHz) and diagnostic ultrasound (1–10 MHz). When a liquid is irradiated by ultrasound, microbubbles appear, grow and oscillate extremely quickly and even collapse violently if the acoustic pressure is high enough. The occurrence of these collapses near a solid surface will generate microjets and shock waves. Moreover, in the liquid phase surrounding the particles, high micromixing will increase the heat and mass transfer and even the diffusion of species inside the pores of the solid (Contamine et al., 1994).

The use of power ultrasound in leather processing in order to improve the efficiency of the process as an eco-friendly approach has been studied and reviewed in detail (Sivakumar and Rao, 2001; Sivakumar et al., 2009c). Recently, the use of ultrasound in the extraction of vegetable tannins (Sivakumar et al., 2007, 2009a) as well as beet dye extraction and natural dyeing of leather (Sivakumar et al., 2009b) has been reported by us. Ultrasound has been used as a process intensifier for various unit operations in leather processing such as leather dyeing, essentially to enhance the diffusion of chemicals through skin/leather matrix. In the case of natural dye extraction, ultrasound is used as a tool for enhancing mass transfer of coloring matter from natural plant material and transport to the solvent medium. The similarity is that ultrasound facilitates the transport processes in both the cases. Hence, ultrasound technique could be beneficial for extraction of natural dyes and subsequently for leather dyeing also.

In this regard, the present paper aims at screening the locally available natural dye yielding plants for dyeing purpose. The effect



Fig. 1. Schematic diagram of variable power output ultrasonic probe.

of power ultrasound in the extraction of natural dyes has been studied.

#### 2. Experimental methods

#### 2.1. Experimental setup

Ultrasonic extraction experiments were performed using ultrasonic probe (VCX 400, Sonics and Materials, USA, 20 kHz and 0–400 W) in a glass vessel with provisions to set required output power and time as shown in Fig. 1. Control experiments were performed with the help of a magnetic stirrer, which had provisions to control temperature.

#### 2.2. Materials and methods

#### 2.2.1. Natural dye materials

Natural dye bearing plant materials such as Green wattle bark, Marigold flowers, Pomegranate rinds, 4'o clock plant flowers and Cocks comb flowers were used. These plant materials were collected fresh from our CLRI Institute garden. The photographs of these materials are shown in Fig. 2a–e, respectively.

#### 2.2.2. Extraction using magnetic stirring (control experiment)

The color bearing plant samples were collected fresh from our CLRI Institute garden and separated into individual petals with average size of 1 cm and used for the experiments. Typically 1 g of sample was taken and 50 ml distilled water was added in a glass beaker in order to keep the plant materials along with ultrasound tip fully immerse in solvent. The beaker was covered using aluminum foil to prevent loss of solvent by evaporation. This beaker was stirred magnetically for 3 h. In order to have the fair comparison with ultrasound system, where ultrasonic bath temperature is around 45 °C without external heating, the temperature of the extraction bath for control process was also maintained at 45 °C. This would also provide idea about improvements with ultrasound extraction other than temperature induced effects of ultrasound. Extract samples were taken at every 30 min and the optical density was determined with the help of UV-VIS spectrometer. At the end of 3 h, the yield and extraction efficiency of each sample was determined by gravimetric method. The extract was tightly closed and stored at low temperature for future reference.

Table 1
Natural dve vielding plants used in the present study.

S. no.	Botanical name	Common name	Parts used	Color
1	Acacia decurrens	Green wattle	Bark	Dark brown
2	Tagetes erecta	Marigold	Flowers	Yellow
3	Punica granatum	Pomegranate	Rind	Yellow
4	Mirabilis jalpa	4'o clock plant	Flowers	Pink
5	Celosia cristata	Cocks Comb	Flowers	Red

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