ISSN 1996-3416

International Journal of Chemical Technology

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International Journal of Chemical Technology 4 (2): 57-65, 2012
ISSN 1996-3416 / DOI: 10.3923/ijct.2012.57.65

Improved Extraction of Natural Blue dye from Butterfly Pea using Microwave Assisted Methodology to Reduce the Effect of Synthetic Blue Dye

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ABSTRACT

Traditionally, extraction of natural dyes with aqueous extraction method involve several hours of extraction time. A rapid and improved extraction technique should be introduced especially to textile dyers for synthesis of natural dyes in a shorter time. This study demonstrates the microwave irradiation as a new technique to extract colorants from a selected flower, i.e. butterfly pea which can be found abundantly in India. Colorant from this flower is extracted at different elevated times, from 10 sec up to 2 min using microwave technique and the extracts obtained are compared to those obtained by aqueous extraction method at 30 min to 3 h. The color strength and yield of dye extracts is analyzed using UV-Visible spectrophotometer. It is observed from the experimental results that the extraction using microwave techniques gives better results than the conventional aqueous extraction methods.

Key words: Natural colourant butterfly pea flower, aquoues extraction, microwave irradiation method for extraction, UV-visible spectrophotometer

INTRODUCTION

Until the unexpected invention of widely available and cheaper synthetic dye "Perkin Mauve" in 1856 (Parkes, 2003), people are using few natural sources like stem, bark, leaves, roots and flowers to get different colors i.e. yellow, orange, blue, red, green, brown, grey etc., for dyeing the clothes. But due to increase environmental pollution and health hazards associated with synthetic dye (Ali and Muhammad, 2008; Arunachalam and Annadurai, 2011; Atmani *et al*., 2009; Bhatnagar and Jain, 2006; Chowdhury and Saha, 2010; Dutta and Basu, 2012; Ghribi and Chlendi, 2011; Jayarajan *et al*., 2011; Rajendran *et al*., 2011; 2012; Rajeswari *et al*., 2011; Saha *et al*., 2012), people have forced to conscious about the alternative preparation of natural dyes. Natural dyes are obtained from any part of plants and are eco-friendly i.e., they do not create any environmental problems at the stage of production or use, maintains ecological balance and have a beautiful attractive shades. The use of natural dyes replaces and minimizes significantly on the amount of toxic effluent resulting from the dye process (Saha and Datta, 2008; Sinha *et al*., 2012).

The extraction of dye from different natural sources can be used in place of synthetic dye in textile industry. Beside the application in textile industry, the dyes can also be used in the

coloration of food industry, for preparing herbal gulal and making different colorful candles. The advantages of natural dyes are cost effective, renewable and non-carcinogenic in nature, no disposal problems and have no allergic reaction on skin.

Traditionally most textile dyers employed aqueous extraction method to extract natural dyes from plant species. Unfortunately, this method consumed several hours of extraction time. It is therefore important to utilize most efficient extraction method in textile dyeing (Saha and Datta, 2008). As the growing popularity of textile industry has led to vigorous come back of natural dye in recent years (Raja and Thilasavathi, 2011), it seems to be a greater need for faster and energy saving technology for the synthesis of these dyes. Although, today's production plants for dyes are technically very advanced due to modernization but despite of chemical and technical improvements there has always been threats to the environmental pollution and energy depletion. Minimization of the environmental pollution and usage of minimal energy are the main issue now a day. In this regard non conventional energy resource like microwave (Majetich and Hicks, 1995; Sinha *et al.*, 2012) has been used extensively in the recent times. Since, Microwave has recently become extremely popular to increase organic reactions by providing high heat efficiency, remarkable rate enhancement and dramatic reduction in reaction times, considered using microwave irradiation in improving the extraction of dye from flower (Mansour and Gamal, 2011).

For the purpose of this study butterfly pea (indigo blue colour) has been chosen as natural source for dye extraction. The aim of the present study was to show a comparative study for extraction of blue dye from butterfly pea using conventional techniques and microwave technique.

MATERIALS AND METHODS

Butterfly pea (*Clitoria ternatea*) is a perennial creeper plant to the family Fabaceae. The flowers are available almost throughout the season (Fig. 1). The most striking characteristic about this plant are its deep blue flowers. Major flavonol glycosides, 3-O-(2"-O-alpha-rhamnosyl-6"-O-malonyl)-betamalonyl)-beta-glucoside and 3-O-(2",6"-di-O-alpha-rhamnosyl)-beta-glucosid were isolated from the blue- flowered petals (Kogawa *et al.*, 2006; Tantituvanont *et al.*, 2008; Terahara *et al.*, 1998). The flowers also contain minor delphinid glycosides, 3-O-b-glucoside, 3-O-(2"-O-a-rahmnosyl-6"-O-malonyl)-b-glucoside of delphinidin (Kogawa *et al.*, 2006). Eight

Fig. 1: Butterfly pea used in this study

anthocyanins (ternatins C1, C2, C3, C4, C5 and D3 and preternatins A3 and C4) were also isolated from flowers (Terahara *et al*., 1996; 1998). Six ternatins from the flowers were partly characterized as highly acylated dephinidin (Terahara *et al*., 1990). *Clitoria ternatea* flowers also contain little calcium (1.9 mg 100 g^{-1}) analyzed using atomic emission spectroscopy (Laurena *et al*., 1994).

Aqueous extraction method: The freshly collected flowers petals with average size of 1 cm were used for the experiments. Different amount of samples (0.1, 0.2, 0.5, 1 and 2 gm) were taken in each Erlenmeyer flask (250 mL) and 50 mL water was added in each and kept into the hot air woven at different temperatures (60, 70, 80, 90°C) and at room temperature. Extract samples were taken at different time intervals (30, 45, 60, 90, 120 and 180 min), filtered and dried. The optical density of the sample was determined with the help of UV-VIS spectrophotometer (HITACHI MODEL NO 2800) and the total weight of the extract dye per gram of the butterfly pea was determined.

Microwave extraction method: Extraction procedure was performed using microwave irradiation method. Different amount of freshly collected sample (0.1, 0.2, 0.5, 1 gm) were weighed and transferred into 100 mL beaker and to it 50 mL of distilled water was poured. The effect of microwave time on the yield of colorant was examined at different time intervals of 10, 30, 50, 60, 75, 90 and 120 sec with the different extraction power (330, 600, 800 w).

Like aqueous extraction procedure, all extracts were filtered and dried. The optical density was measured by the UV-VIS spectrophotometer (HITACHI MODEL NO 2800) and the total weight of the colorant extract per gram of the butterfly pea was determined.

Characterization of the dye: Scanning Electron Microscopy (SEM) was used to study the surface morphology of the adsorbent. SEM studies were carried out using a scanning electron microscope (Model Hitachi S-3000N) at an electron acceleration voltage of 20 kV. Prior to scanning, the adsorbent was coated with a thin layer of gold using a sputter coater to make it conductive.

Fourier Transform Infrared Spectroscopy (FTIR) analysis: Functional groups present on the biosorbent surface can be identified by Fourier transform infrared (FTIR) spectroscopy as each group has a unique energy absorption band. Therefore, an FTIR spectrum of the biosorbent was recorded. Initially, the ground tamarind seeds were dried overnight at 333±1 K in an oven drier, to remove the water in preparation for the FTIR analysis. Then, 0.001 g of dried sample was mixed with 0.5 g of spectroscopic grade potassium bromide powder in an agate pestle and mortar. The powder was then compressed into a thin KBr translucent disk under a pressure of 100 kg cm^{-2} for 8 min with the aid of a bench press. FTIR spectrum was then recorded with Fourier transform infrared spectrometer (Perkin-Elmer Spectrum BX-II Model). The sample compartment was continuously purged with dry air to minimize water vapor and carbon dioxide interference. FTIR spectra were recorded in the wavenumber range 4000-500 at 4 cm^{-1} spectral resolution.

RESULTS AND DISCUSSION

Blue dye from different amount of butterfly pea was extracted using microwave irradiation to help considerable improvisation in both the time and yields (Table 1). It was observed from the experimental results that the extraction of dye from butterfly pea employing microwave irradiation

Table 1: A comparative study between extractions of dye using butterfly pea flower

Wight of the flower (g)	Conditions	Conventional	Microwave
0.1	Reaction time	3 h	2 min
	Total dye in solution (mg L^{-1})	515	617
	Absorbance	0.2169	0.2847
	Product condition	Unclean	Clean
0.2	Reaction time	3 h	2 min
	Total dye in solution (mg L^{-1})	691	1135
	Absorbance	0.3482	0.4783
	Product condition	Unclean	Clean
0.5	Reaction time	3 h	2 min
	Total dye in solution (mg L^{-1})	1315	1890
	Absorbance	0.7775	1.230
	Product condition	Unclean	Clean
1	Reaction time	3 h	2 min
	Total dye in solution (mg L^{-1})	2022	3985
	Absorbance	1.373	4.605
	Product condition	Unclean	Cclean

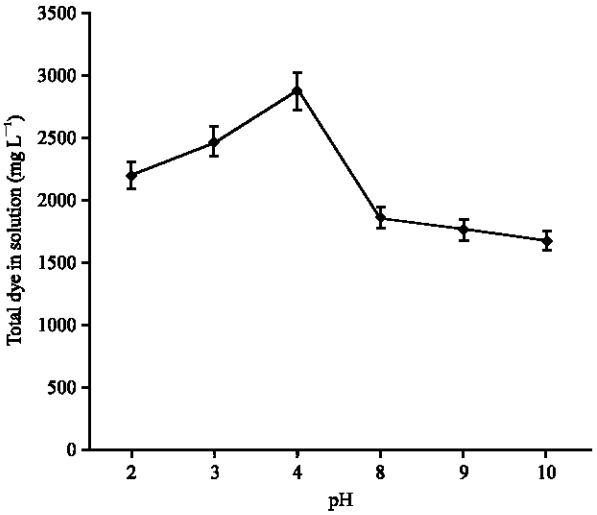

Fig. 2: Extraction of dye from 1 g butterfly pea at different pH using microwave irradiation (irradiation time = 1 min)

was completed in 2 min with 4.605 mg L^{-1} dye extract in solution. On the other side, using the conventional method (aqueous extraction method) it required 3 h for yielding the same blue dye as microwave extracted dye. This comparative study revealed that not only the reaction time reduced from 3 h to 2 min but also the yield of extract material was better in the microwave method. When microwave irradiation was used, the reaction mixture was also found cleaner than the conventional method. As the time of extraction increased, the extraction of dye increased in both the processes but the amount of dye extraction was higher in microwave process at same time intervals.

Effect of pH: From Fig. 2, it was observed that the extraction of dye from butterfly pea flower was a function of pH. The effect of pH on dye extraction was analysed over a pH range 2 to 10 (Fig. 2).

As can be observed from Fig. 2, the dye extraction increased with increasing the pH of solution from 2 to 4 and then after it gradually decreased. It was found that if the aqueous medium was slightly acidic, then extraction of dye in water increased as the efficiency of extraction process increased in presence of weakly acidic medium rather than in basic medium for butterfly pea dye. The effect of pH on the extraction of butterfly pea solution was determined by spectrophotometric analysis). Scan spectrum showed different λ_{max} values at different pH. The absorbance at 550, 574, 627, 629 and 670 nm was monitored for the extract solutions at a pH value of 2, 4, 6, 8 and 10, respectively. In acidic pH solution, the color displayed a red color; but in alkaline pH solution, the color changed to greenish blue. The change in colour and extraction rate of butterfly pea solution depends on the change in equilibrium of four anthocyanin species in its petals according to the prevailing pH (Brouillard, 1982; Saha and Datta, 2008; Tantituvanont *et al*., 2008). The change in the colour intensity at different pH was due to the equilibrium of four anthocyanin species present in the dye. At lower pH, the red colour signified the presence of anthocyanins and in increasing pH, the colour intensity transformed to blue colour due to the presence of quinonoidal base and the yellow colour was for the chalcone.

Effect of extraction power: The effect of power was also an important factor during dye extraction. In order to make a comparative study of natural dye extraction from butterfly pea in presence of different powers (330, 660 and 800 w) of microwave irradiation, the amount of butterfly pea flower and pH was fixed at 1 gm and pH 7, respectively. Over the examined range, it was observed that the rate of dye extraction increased as the power rose (Fig. 3). The reason behind the phenomenon was that as the power increased, it broke the petals structure more efficiently and the dye inside the petals extracted in solution.

The visible spectrum of colorant extracted by microwave irradiation method at 2 min was compared to that obtained by Aqueous extraction method at 180 min. Figure 4 showed that the maximum absorbance obtained at λ max 575 nm for microwave extract which is higher than the aqueous extraction method.

Characterization of butterfly pea dye: The surface structure of the dye materials is uneven, irregular and porous. Further, the pores on the surface of the dye molecules are highly

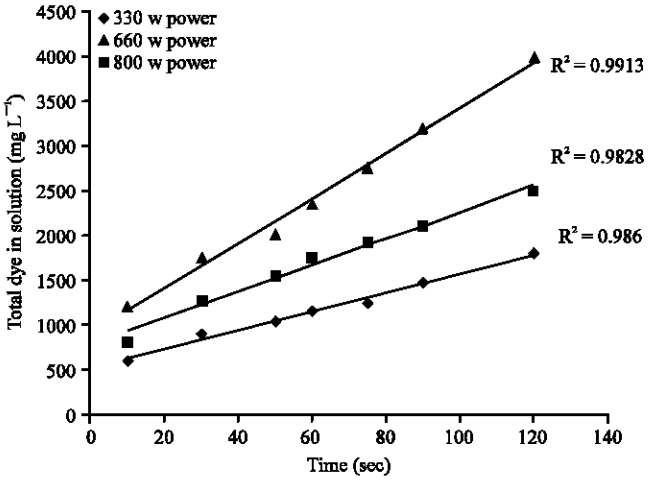

Fig. 3: Extraction of dye from 1 g butterfly pea at different power using microwave irradiation

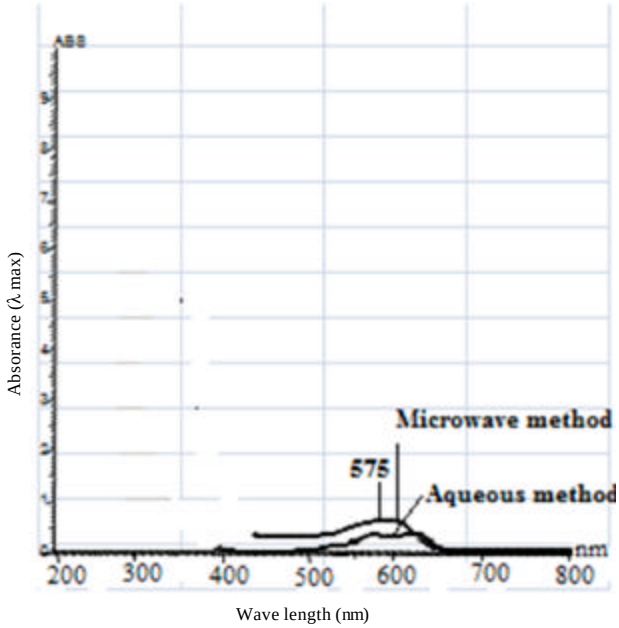

Fig. 4: Spectrum of natural colorant extracted by microwave irradiation method and aqueous extraction method

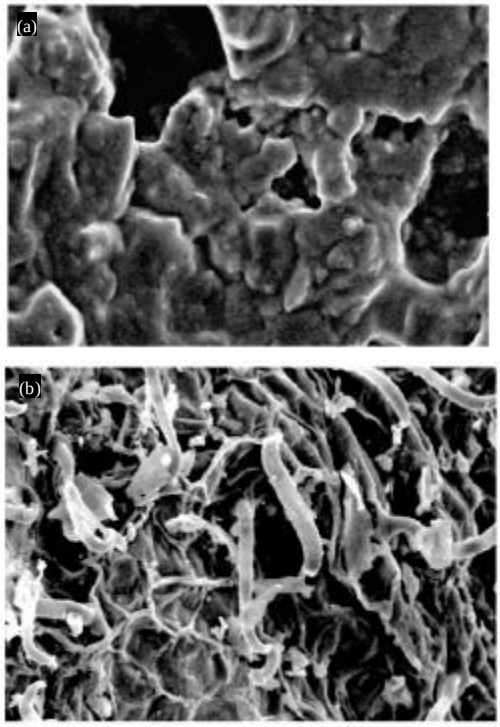

Fig. 5(a-b): Scanning electron micrograph of butterfly pea dye by (a) aqueous extraction method and (b) microwave assisted extraction method

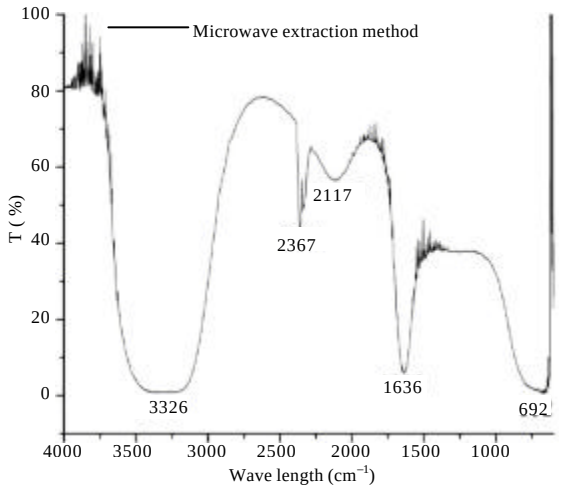

Fig. 6: FTIR spectra of extracted dye from Butterfly pea using Microwave assisted extraction method and aqueous extraction method

heterogeneous. The heterogeneous pores and cavities provide a large exposed surface area. Surfacemorphology of butterfly pea dye extracted by both aqueous extraction method and microwave irradiation method, was studied with the scanning SEM (Fig. 5a, b). These figures revealed that the surface texture of the microwave extracted butterfly pea dye is slightly changed from the aqueous extraction butterfly pea dye.

The FTIR spectral analysis is important to identify the characteristic functional groups on the surface portion of blue petal of butterfly pea flower which are responsible for microwave extraction of blue dye (Fig. 6). The FTIR spectral analysis of butterfly pea dye reveals distinct peaks at 3326, 2367, 2117, 1636 and 692 cm^{-1} for the following chemical characterization. The broad and strong peak at 3326 cm^{-1} indicates the presence of aliphatic C-H stretching from the CH_2 group. The 0-H stretching vibration could be assigned to the band that appeared at 2367 cm^{-1}. Absorbance at 2117 cm^{-1} is due to C≡C stretching vibration. The strong peak at 1636 cm^{-1} shows the presence of -the C = O stretching or N-H vibration of -COOH group or amide groups. The band at 692 cm^{-1} arises from b-glucosidal linkage. Hence, FTIR spectral analysis shows the presence of chemical groups like -CH, -OH, C≡C and -C = O in the extracted butterfly pea dye.

CONCLUSION

The most important features of the work are being enhanced reaction times and formation of cleaner products. People have been interested in looking environmentally safer and cost effective technology for producing the natural blue dye from butterfly pea. This microwave reaction is especially performed to work with open vessels, thus to avoid the risk of high pressure development. From the experimental results it was observed that using microwave techniques, the time of extraction was reduced from 3 h to 2 min for the same amount of dye extraction.

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