

## Ultrasound-microwave assisted extraction of natural colorants from sorghum husk with different solvents



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### ARTICLE INFO

#### Keywords:

Sorghum husk  
Ultrasound-microwave assisted extraction  
Solvent  
Dyeing  
Color

### ABSTRACT

Ultrasound-microwave-assisted (UMA) extraction method with blended solvent 70% ethanol/30% water with HCl significantly improved extraction of biocolorants from sorghum husk. Sorghum is a staple food in many parts of the world. As the demand for the grain soars, there is an equivalent increase in quantity of sorghum husk generated. Sorghum husk is a promising source for natural functional dye. However, the phenolic colorants in sorghum husk are bound to the cell wall and are difficult to extract. This paper is focused on improving the extraction of natural dye from sorghum husk and investigating the effect of the extraction methods and solvents on the dyeing properties of sorghum husk extracts (SHE). UMA extraction method with blended solvent 70% ethanol/30% water with HCl (1 ml HCl per 100 ml) had a SHE yield of 16.7%, which was 3.6 times the yield of 4.6% by conventional shaking (CS) method with water as solvent. SHE extracted by the UMA method had high contents of apigeninidin as well as luteolinidin and higher thermal stabilities than SHE extracted with water. SHE with different solvents produced different shades when dyeing wool and cotton fabrics. SHE by 70% ethanol/30% water with or without HCl had similar dye strength and significantly higher dye strength compared to SHE extracted by water. The dyed wool and cotton fabrics had good colorfastness to laundry, crocking and light. UMA extraction with blended solvent 70% ethanol/30% water with HCl was an efficient method to extract dye from sorghum husk and provided the potential for industrial application. The use of sorghum husk as a source of industrial dye will significantly add value to the sorghum plant and reduce the disposal of sorghum husk.

### 1. Introduction

There has been an increasing tendency towards the use of sustainable and environmentally friendly natural dyes (Shahid-Ul-Islam and Sun, 2017). The pursuit of dyes and coloring matter from nature for textiles is on the rise due to the awareness of the adverse effect of some synthetic dyes on the environment and human health. It was reported that an estimated 10–15% dye was lost in the effluent during the dyeing processes (Iqbal and Ashiq, 2007). The use of environmentally benign dyes and chemicals is a way to control the effect of harmful effluents (Sivakumar et al., 2009). In addition, dyes from nature are of interest for their functional properties like deodorizing effect, antimicrobial, antioxidant and UV protection on textiles products. (Shahid-Ul-Islam and Sun, 2017).

The growing demand for natural dyes necessitates extraction

techniques which are more effective and efficient. A solvent-extraction procedure is commonly used in industry. An efficient solid-liquid extraction method for natural colorants will enhance its usage in the textiles and food industries. Some studies have provided extraction methods that are compatible with the environment and safety concerns. Among the new dye extraction techniques, the use of pressurized fluids, ultrasound and microwave techniques seem to be the most favorable (Duval et al., 2016).

Sorghum is a cereal crop grown in the tropical, subtropical and arid regions and happens to be the fifth leading crop in terms of production after wheat, maize, rice, and barley with an annual production of about 57 million tons globally (Awika et al., 2004). Sorghum is a staple food, livestock feed and has been used to produced bioethanol in many parts of the world (Kim and Dale, 2004). Sorghum is a rich source of various phytochemicals such as tannins, phenolic acids, 3-deoxyanthocyanidins

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and anthocyanins (Dykes et al., 2009). These phytochemicals have health benefits such being anti-carcinogenic, anti-inflammatory, antioxidant, fighting cardiovascular disease and obesity (Awika and Rooney, 2004; Barros et al., 2012; Dykes et al., 2013; Wu et al., 2013). Hence, there is an increased interest in growing sorghum (Dykes et al., 2013). Sorghum husk is a byproduct which is easy to obtain, inexpensive and readily available biomass.

The abundant sorghum husk could be a promising source for natural functional dye (Hou et al., 2017). However, the cell wall bound phenolic compounds are not easily extracted from the hull of cereals using conventional shaking (CS) extraction method with 70% (v/v) ethanol/30% water as solvent, sorghum husk extracts (SHE) had a yield of 6.15% based on weight of powdered sorghum husk (Hou et al., 2017) and hence an effective alternative is needed for maximum polyphenol extraction. In industry, phenolic compounds extraction involves solvent-extraction procedures of which water and ethanol are more environment-friendly solvents (Kayodé et al., 2012). Barros et al. (2013) significantly improved phenolic compounds extraction at temperatures above 100 °C with water and ethanol/water from sorghum bran. There was a significant improvement in extraction of biocolorant from sorghum leaf sheaths with the addition of HCl to ethanol/water solvent. The crude extract from the leaf sheaths had high antioxidant capacity (Barros et al., 2013; Kayodé et al., 2012). The ultrasound-assisted extraction technology with ethanol/water solvent was employed to obtain high extraction yields of phenolic compounds from sorghum husk (Hou et al., 2016). Microwave-assisted procedure was applied to extract phenolic acids from sorghum bran (Chiremba et al., 2012). Akogou et al. (2018) suggested that future research should focus on the improvement of the extraction methods for sorghum biocolorants.

Ultrasound-assisted extraction is an emerging potential technology that can accelerate heat and mass transfer and has been successively used in the extraction field (Chemat et al., 2017a; Sivakumar et al., 2011). Sivakumar et al. (2009) undertook a comparative colorant extraction from beetroot using ultrasound and static/magnetic stirring. Colorant extraction from beetroot was significantly improved using ultrasound. An earlier research had indicated that ultrasound-assisted extraction was effective in extracting oil from pomegranate seed and this resulted in few changes in fatty acid composition (Barizão et al., 2015).

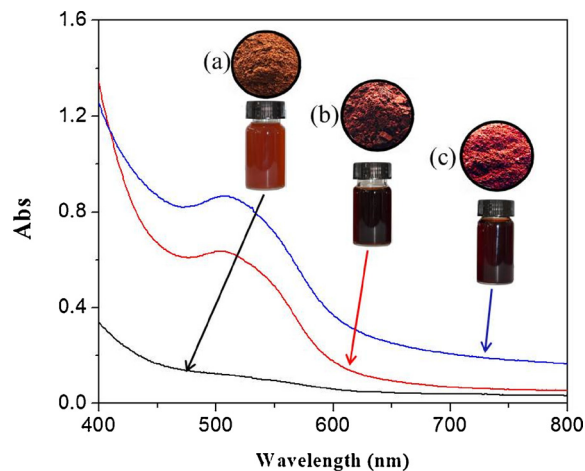
The application of microwave irradiation proves to be a rapid and an improved method of extracting dye from pomegranate peels and also significantly reduces extraction time (Sinha et al., 2012). Microwave presents the disadvantage of inhomogeneous heating (Bonrath, 2004). Therefore, ultrasound-assisted extraction in combination with microwave-assisted extraction, i.e., ultrasound-microwave-assisted (UMA) extraction will be one of the most promising hybrid techniques for fast and efficient extraction (Chemat et al., 2017b). The simultaneous use of microwave and ultrasound in a single reactor has been implemented in order to combine the effects of enhanced energy with improved matter transportation (Barrera Vázquez et al., 2014; Leonelli and Mason, 2010). The earlier studies reported that UMA extraction had better yield for the total anthraquinones from the stems and leaves of *Heterophyllaea pustulata* Hook.f. (Rubiáceae) (Barrera Vázquez et al., 2014), juglone from walnut green husk (Xu et al., 2016; Yin et al., 2016), polysaccharides from *Cornus officinalis* (Yin et al., 2016). However, the great potential of this hybrid technique has not yet been adequately exploited (Chemat et al., 2017b).

This paper is focused on improving the extraction of natural dye from sorghum husk and also investigates the effect of extraction methods and solvents on the dyeing properties of SHE. The results could promote the industrial application of the biocolorants from sorghum husk in the textile industry.

**Table 1**  
SHE yield for different extraction methods and solvents.

| Extraction solvents                   | yield of SHE  |                | % improvement due to ultrasound-microwave assistance (B-A)/A × 100 |
|---------------------------------------|---------------|----------------|--|
|                                       | CS method (A) | UMA method (B) |  |
| Water                                 | 4.6 ± 0.15    | 5.6 ± 0.55     | 21.7   |
| 70% ethanol/<br>30% Water             | 5.6 ± 0.60    | 10.6 ± 0.50    | 89.3   |
| 70% ethanol/<br>30% Water<br>with HCl | 10.4 ± 0.30   | 16.7 ± 0.60    | 60.6   |

Note: Extraction condition for CS method was 80 °C for 60 min and UMA method was 55 °C for 20 min.



**Fig. 1.** The absorbance curves of SHE liquid and SHE powder extracted by UMA method with different solvents (a) water; (b) 70% ethanol/30% water; (c) 70% ethanol/30% water with HCl.

**Table 2**  
The UV–vis absorbance of the solution extracted from sorghum husk by UMA method and 70% ethanol/30% water with HCl as blended solvent under different extraction conditions.

| Extraction time (min) | Extraction temperature (°C) | Absorbance at $\lambda = 281$ nm | Absorbance at $\lambda = 400$ nm | Integration of absorbance in visible region (from $\lambda = 400$ to 800 nm) |
|-----------------------|-----------------------------|----------------------------------|----------------------------------|--|
| 10                    | 65                          | 1.00                             | 0.32                             | 260.49   |
| 20                    | 65                          | 1.05                             | 0.36                             | 259.20   |
| 20                    | 55                          | 1.25                             | 0.47                             | 327.75   |
| 20                    | 45                          | 1.23                             | 0.45                             | 273.33   |
| 30                    | 65                          | 0.81                             | 0.19                             | 179.02   |

## 2. Experimental

### 2.1. Materials and chemicals

The dried sorghum husks were purchased from Changling, China. Worsted wool fabric was purchased from Wuxi Xiexin Textile Company, China. The wool fabric had the following characteristics: Gabardine, warp and weft 17 × 2 tex, warp density 402 threads per 10 cm, weft density 225 threads per 10 cm, weight 237 g/m<sup>2</sup>. Bleached cotton fabric was purchased from Luolai Lifestyle Technology Co. Ltd. and had the following characteristics: satin weave, warp and weft 14 tex, warp density 570 threads per 10 cm, weft density 335 threads per 10 cm and weight 115 g/m<sup>2</sup>.

Acetic acid, hydrochloric acid (HCl), sodium carbonate, sodium hydroxide, ethanol, aluminum potassium sulfate (KAl(SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O)

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