

NATURAL DYE YIELDING POTENTIAL AND COMPOUNDS OF SELECTED VEGETABLE RESIDUES BELONGING TO BRASSICACEAE: AN APPROACH TOWARDS SUSTAINABILITY

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Abstract

The cruciferous vegetables are well known for their ethnobotanic and economic importance throughout the world. Traditionally, the members of Brassicaceae are used as vegetables, food plants, ornamentals as well as source of oil and natural dyes. Though there are evidences regarding the historic use of Brassicaceae plants for coloring of various items but information for utilization of these in modern textile dyeing is negligible. Current study is concerned with the utilization of residual material of *Brassica oleracea* L. var. *capitata* (cabbage), *Brassica oleracea* L. var. *botrytis* (cauliflower) and *Brassica rapa* L. var. purple top (turnip) to achieve maximum dye yielding potential as well as determine their phytochemical nature. Results revealed that colorants of varying shades could be extracted from above mentioned plant residues using different extraction media (acidic, alkaline, aqueous and organic). Variety of color shades including light brown, brown, yellowish green, yellow, dark green, creamy white, light green, olive green and dark brown could be produced from these plant residues using eco friendly bio as well as chemical mordants. The analysis of these plant residues revealed the presence of flavonoids, tannins, alkaloids, saponins, carbohydrates, sugars and glycosides. Stability of these vegetable residues based colorants in terms of fastness properties including wet rubbing, washing, dry rubbing and light fastness proved to be good to excellent.

Key words: Vegetable dye, Cotton fabric, Secondary metabolites, Fastness properties.

Introduction

Plants, the potential source of varying components including primary and secondary metabolites are of great importance for humans and all other living organisms (Manoharachary & Nagaraju, 2016). The use of plants in different civilizations for food and dyeing purpose as well as cultural, religious activities has been well documented (Dogan *et al.*, 2003; Liu *et al.*, 2014; Salinitro *et al.*, 2017; Yilmaz *et al.*, 2018). For the survival of mankind, importance of plants has well been emphasized. Almost four hundred plants are being utilized as vegetables by human beings (Kays & Dias, 1995; Liu *et al.*, 2001). These plants contain nutrients, metabolites and natural colorants (Hounsome *et al.*, 2008; Pagare *et al.*, 2015). Natural dyes are esthetically important and considered eco friendly competitors of hazardous synthetic ones (Mir *et al.*, 2019; Batoool *et al.*, 2019). Different plant species, algae, insects, minerals, microbes and lichen are well known sources of natural dyes (Baaka *et al.*, 2017; Meryemoglu, 2018; Azeem *et al.*, 2019; Shaheen *et al.*, 2019).

Exploration and sustainable utilization of flora is an important domain of Economic Botany (Baydoun *et al.*, 2017; Junsongduang *et al.*, 2017) with particular importance in ecological zones of rich biodiversity of Pakistan (Ahmad *et al.*, 2008). Most of the work has been conducted to explore nutritional or medicinal value of plants. The vegetables, especially the non edible part has huge potential to be used for value addition, such as source of natural dyes. The prodigious features like eco friendly and biodegradable nature has ignited the status of plant dyes (Batoool *et al.*, 2013; Raza *et al.*, 2018). Previous studies revealed that only few plant species have

been explored regarding their dye yielding potential (Kasiri & Safapour, 2015). To fulfill the increasing demand of industry, chemical dyes are utilized on large extent. Utilization of synthetic dyes on large scale has crumbled the eco balance by imposing negative impacts on aquatic life and human health (Saravanan *et al.*, 2014; Gita *et al.*, 2017).

The plants belonging to Brassicaceae, mostly represented in temperate and sub tropical regions of the world (Gidik *et al.*, 2016) are well adapted to climate of Pakistan, where 92 genera and 250 species of this family have been reported (Perveen *et al.*, 2004). Brassicaceae plants have great ethnobotanical importance and traditionally utilized as food as well as fodder (Romano *et al.*, 2013; Rahman *et al.*, 2018) and source of natural colorants (Riyaz & Thaseen, 2017; Haddar *et al.*, 2018). A number of vegetables of this family have been reported to contain phenolics and antioxidants (Cartea *et al.*, 2011; Collett *et al.*, 2014), polyphenols (Ferrerres *et al.*, 2005; Jaiswal *et al.*, 2012), glucosinolates (Kusznierewicz *et al.*, 2008), tocopherol, proanthocyanidins, alkaloids, ascorbic acid and carotenoids (Jahangir *et al.*, 2009; Guyria *et al.*, 2015). The leaves having different phytochemicals (Saad *et al.*, 2016) are used to cure diseases (Engel *et al.*, 2002; Sasaki & Takahashi, 2002; Schonhof *et al.*, 2004).

Considering the current scenario, there is a dire need to explore maximum flora, especially non edible part of vegetables to get its hidden dye wealth and ethno botanical significance. Current study is primarily focused on the utilization of surplus material of vegetables as dye source as value addition to strengthen the economy. The objective of the study is to compare the relative dye yielding potential of selected vegetable residue belonging to Brassicaceae for fabric dyeing.

Materials and Methods

Collection of vegetable residual material: The vegetable residual material of plants belonging to Brassicaceae was collected from vegetable fields of Ayub Agricultural Research Institute, Faisalabad during winter growing season (Fig. 1). Residual materials of following vegetable plants belonging to Brassicaceae were evaluated for their colorant yielding potential to be used for textile dyeing.

- 1) *Brassica oleracea* L. var. *capitata* (cabbage)
- 2) *Brassica oleracea* L. var. *botrytis* (cauliflower)
- 3) *Brassica rapa* L. var. purple top (turnip)

The residual material included the non-edible leftover parts such as aboveground stem and leaves of the selected plants. Dried vegetable residual materials were grinded into fine powder. The phytochemical tests, extraction of

natural colorant and dyeing of cotton fabrics with the extracted colorants were performed in Economic Botany lab at department of Botany Government College University Faisalabad, Pakistan.

Natural colorant extraction: The natural colorants from vegetable residual material were extracted using different extraction media (Fig. 2). The media used to extract colorants from selected vegetable residues included aqueous (water), alkaline (sodium hydroxide and potassium hydroxide), organic (methanol, ethanol and methanolic potassium hydroxide) and acidic (hydrochloric acid, acidified ethanol and acidified methanol). Extraction was carried out by boiling 4.0 g of each of dye powder in above extraction media separately for 40 minutes at hot plate. After boiling, extracts were filtered and tested for dyeing of fabric. The treated fabrics were thoroughly washed before drying and examined under Spectra flash SF 600 for the determination of color strength characteristics.



Fig. 1. The selected vegetables growing in the field area (a=Cabbage leaves, b= Cauliflower leaves, c= Turnip leaves).

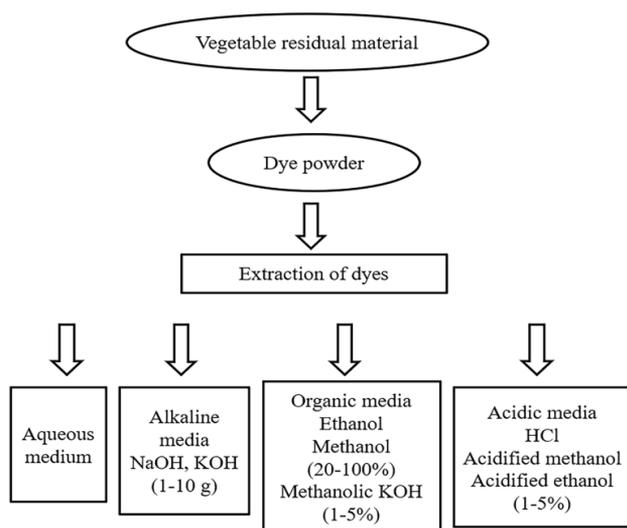


Fig. 2. Schematic diagram of vegetable residual based colorant extraction using varying extraction media.

Mordanting and fastness properties: The selected vegetable residual material based colorant treated cotton fabrics were reacted with herbal mordants such as dried henna leaves, pomegranate rind, turmeric powder, golden

shower bark and onion peel in comparison with chemical mordants such as tannic acid, potassium dichromate, copper sulphate and iron sulphate. Color strength, in terms of K/S values of mordanted fabrics was recorded by using Spectra flash SF- 600. Colorant stability of mordanted fabrics against light, washing and rubbing was determined following standard methods, such as ISO 105 B02 for light fastness, ISO105-C03 for washing fastness and ISO 105 X-12 for rubbing fastness.

Phytochemical analysis: Analysis of crude vegetable leftover material was performed by boiling dye powder in sterile water. Dye extract was filtered using Whatman, s No.1 filter paper. The filtrate was centrifuged at 2500 rpm for 15 minutes. Dye extract was used for detection of phytochemicals (Savithamma *et al.*, 2011; Soni & Sosa, 2013). Presences of alkaloids were estimated using Mayer's reagent. Tannins were determined by Braymer's test, flavonoids by alkaline reagent test and phenolics by ferric chloride test. Qualitative determination of saponins was carried out by foam test, terpenoids by Salkowski test, carbohydrates by Molisch's test and sugars by Benedict's test. Analysis of variance (ANOVA) technique was employed using completely randomized design. The CoStat computer software was utilized for statistical analysis.

Results

The statistical manipulation of data indicated that different extraction media produced varying effects on natural colorant yield of selected vegetable species (Tables 1-4). Data given in tables 1, 2, 3 and 4 indicated that 3% acidified methanolic solution for cabbage leaves, 100% methanolic solution for cauliflower leaves and acidified methanol (2%) for turnip leaves were proved to be more suitable extraction media to get higher dye yield. Vegetable residual material yielded different colorant using varying solvents. These colorants gave varying color shades on cotton fabric (Table 5). Presence of varying phytochemicals like flavonoids, alkaloids, glycosides, tannins, saponins, sugars and carbohydrates were recorded in vegetable residual material (Table 6).

The data regarding chemical mordanting of cabbage leaves based dye indicated that 8% FeSO_4 as pre mordant, 6% FeSO_4 as post mordant and 2% turmeric as bio mordant were highly effective levels to produce darker shade of fabric. The cauliflower leaves based dye produced darker shades with 2% FeSO_4 as pre-mordant as well as 8% $\text{k}_2\text{Cr}_2\text{O}_7$ as post-mordant and 4% dried pomegranate peel as bio pre-mordant and 8% turmeric as bio post-mordant. Turnip leaves based dye on cotton fabrics using mordanting agents produced different shades. In chemical mordanting, 8% and 4% tannic acid as pre- as well as post mordants produced darker shades. While, in bio-mordanting, 8% turmeric powder as pre-mordant and 6% onion peel as post mordant gave darker shades.

Color shades of natural dye extracted from each vegetable residual material vary with respect to different extraction media and mordant used (Table 7). Results revealed that varying extraction media produced different color shades such as light brown, creamy white and olive green from cabbage, cauliflower and turnip leaves. Strong relationship between dye and secondary metabolites of vegetable residual material were observed. Upon interacting with bio and chemical mordants, vegetable residual material produced yellow, dark yellow, light brown, dark brown, creamy white, green, yellowish green and olive green shades (Table 5). The bio- mordanting produced comparatively darker shades than that of chemical mordanting (Table 5). Color fastness properties including light, washing and rubbing fastness indicated higher values of vegetable residue based colorants. Upon testing, optimized mordanted fabrics showed maximum resistance to light, detergent as well as to dry and wet rubbing and consequently gave best results in term of fair, good and excellent rating of fastness (Table 8).

Discussion

Secondary metabolites of plants are considered to have a major role in varying color shade production on cotton fabrics (Pagare *et al.*, 2015). Metal salts as chemical mordants and bio mordants are also known to produce variety of color shades upon reacting with varying metabolites. Exploration of natural products is gaining considerable attention of scientists in the current era of industrial dominance (Lai & Chang, 2021). Every year, millions of tons of vegetables are cultivated across the globe and conversion of their left over converted into value added product could be of great importance. The extraction of natural colorant previously

reported from some vegetables, such as black carrot, red carrot, red cabbage, purple cabbage, egg plant skin, sugar beet and onion skin (Parvinzadeh & Kiumarsi, 2008; Almahy *et al.*, 2013; Shukla & Vankar, 2013; Tasneem & maria, 2016; Riyaz & Thaseen, 2017; Haddar *et al.*, 2018; Debnath *et al.*, 2018; Batool *et al.*, 2019; Pucciarini *et al.*, 2019) usually involves the useable parts of the plants. The current study provided an insight to utilize vegetable leftovers to fulfill demand of colorant materials at large scale (Fan *et al.*, 2018). Literature studies revealed that dye color vary with harvesting season, soil properties, plant part used, fresh or dried material used and concentration of colorant (Lambare *et al.*, 2011). Vegetable based dyes provide resistance to dyed fabrics having antimicrobial potential and anti fungal properties (Compean & Ynalvez, 2014).

The results of current experiments showed that comparatively small amount of acidic and organic media proved to be cost effective and environment friendly in term of yielding maximum colorant (Shabbir *et al.*, 2016; Chao *et al.*, 2017). Colorant rating differences as recorded during current study could be explained as complex formation tendency of dye molecules with mordants and fabrics (Hosseinnzhad *et al.*, 2017; Hosen *et al.*, 2021). The weak complex formation resulted the fading of dye in light (Naz *et al.*, 2011). The water soluble dye molecules present in vegetable residual extracts might have dissolved upon washing and either wet or dries rubbing indicating low fastness properties (Haji, 2010; Ahmed *et al.*, 2019).

The versatile nature of phytochemicals (Singh *et al.*, 2018) has highlighted their importance in varying fields (Mallik & Akhter, 2012; Chatatikun & Chiabchalard, 2013; Ashrafudoulla *et al.*, 2016). The different colorant shades obtained from vegetable leftovers extracts after mordanting indicated the varying nature of secondary metabolites present in residual material of vegetables used in current study.

Presence of phytochemicals like, saponins, alkaloids, flavonoids and glycosides in cabbage, cauliflower and turnip leftovers as revealed during phytochemical analysis might be responsible for light brown, creamy white and olive green shades, respectively onto cotton fabric. Many of the phytochemicals historically acted as a colorant and have been utilized in varying domains (Parthasarathi & Lokesh, 2015; Altemimi *et al.*, 2017). Similarly, Haddar *et al.*, (2018) obtained light purple, pink and blue shades on silk and wool fabric using red cabbage as colorant source.

As evident from the results of current research, non edible vegetable parts of cabbage, cauliflower and turnip could be used to develop light brown, creamy white and olive green shades, respectively onto cotton fabric. Furthermore, a variety of color shades of mentioned vegetables based colorants dyed cotton fabric can be produced upon mordanting. The mordanting may yield brown, green, yellow green and dark yellow shades onto cotton fabrics dyed with cabbage leaf extract. Similar shades like dark brown, dark yellow and yellowish green may be produced onto cotton fabrics treated with turnip leftover material. In addition, the use of mentioned vegetable leftover based colorants showed good color strength properties. The most suitable media for extraction of natural colorant from cauliflower, cabbage and turnip were 100% methanol, 3% acidified methanol and 2% acidified methanol, respectively. Among all the three vegetables used in the study cauliflower leftovers yielded colorants with good strength and fastness properties followed by cabbage and turnip.

Table 1. Color strength (K/S) value of cotton fabrics dyed with natural colorant of Brassicaceae vegetable residues using aqueous and alkaline extraction medium.

| Extraction media | Concentration (%) | Cabbage | Cauliflower | Turnip | |
|------------------|-------------------|---------|-------------|--------|------|
| Aqueous | 100% | 0.34 | 0.31 | 0.37 | |
| | NaOH | 1 | 0.85 | 0.50 | 0.28 |
| | | 2 | 0.85 | 0.48 | 0.26 |
| | | 3 | 0.94 | 0.70 | 0.30 |
| | | 4 | 1.04 | 0.69 | 0.26 |
| | | 5 | 1.02 | 0.89 | 0.46 |
| | | 6 | 1.36 | 1.07 | 0.22 |
| | | 7 | 1.17 | 1.09 | 0.29 |
| | | 8 | 0.53 | 1.65 | 0.27 |
| | | 9 | 0.59 | 1.01 | 0.25 |
| 10 | | 0.49 | 1.02 | 0.23 | |
| KOH | 1 | 0.42 | 0.43 | 0.49 | |
| | 2 | 0.56 | 0.58 | 1.26 | |
| | 3 | 0.92 | 0.59 | 0.51 | |
| | 4 | 1.01 | 0.82 | 0.46 | |
| | 5 | 0.81 | 0.87 | 0.58 | |
| | 6 | 0.58 | 0.69 | 0.66 | |
| | 7 | 0.79 | 0.48 | 0.53 | |
| | 8 | 0.62 | 0.45 | 0.53 | |
| | 9 | 0.50 | 0.60 | 0.53 | |
| | 10 | 0.44 | 0.60 | 0.52 | |

Table 2. Color strength (K/S) value of cotton fabrics dyed with natural colorant of vegetable residues extracted in organic medium.

| Extraction media | Concentration (%) | Cabbage | Cauliflower | Turnip |
|------------------|-------------------|---------|-------------|--------|
| Methanol | 20 | 0.42 | 0.97 | 1.15 |
| | 40 | 1.23 | 0.89 | 1.70 |
| | 60 | 3.2 | 1.17 | 2.33 |
| | 80 | 2.86 | 1.29 | 1.47 |
| | 100 | 2.99 | 5.99 | 2.28 |
| Ethanol | 20 | 0.53 | 0.36 | 0.54 |
| | 40 | 0.46 | 0.65 | 0.85 |
| | 60 | 1.17 | 0.58 | 0.93 |
| | 80 | 1.24 | 0.55 | 1.49 |
| | 100 | 0.88 | 1.32 | 0.72 |
| Methanolic KOH | 1 | 1.18 | 0.36 | 1.47 |
| | 2 | 1.42 | 0.49 | 3.06 |
| | 3 | 1.69 | 0.18 | 2.34 |
| | 4 | 2.10 | 0.27 | 2.18 |
| | 5 | 2.04 | 0.20 | 1.54 |

Table 3. Color strength (K/S) value of cotton fabrics dyed with natural colorant of vegetable residues extracted in acidic medium.

| Extraction media | Concentration (%) | Cabbage | Cauliflower | Turnip |
|--------------------|-------------------|---------|-------------|--------|
| Acidified methanol | 1 | 1.66 | 2.84 | 2.58 |
| | 2 | 2.55 | 3.42 | 4.03 |
| | 3 | 3.78 | 1.60 | 3.35 |
| | 4 | 2.99 | 2.47 | 3.33 |
| | 5 | 1.66 | 2.31 | 2.21 |
| Acidified ethanol | 1 | 1.28 | 1.16 | 1.41 |
| | 2 | 2.80 | 1.58 | 1.42 |
| | 3 | 3.20 | 1.24 | 1.12 |
| | 4 | 2.35 | 1.24 | 1.02 |
| | 5 | 2.31 | 1.00 | 1.07 |
| Acid | 1 | 1.00 | 0.97 | 0.81 |
| | 2 | 1.34 | 1.46 | 1.75 |
| | 3 | 0.70 | 1.34 | 1.58 |
| | 4 | 0.45 | 0.50 | 1.67 |
| | 5 | 0.19 | 0.42 | 1.03 |

Table 4. Mean square values from ANOVA indicating the performance of different media regarding extraction of colorants from vegetable leftovers.

| Plant species | | Degree of freedom | Mean square value |
|--------------------|-------------------|-------------------|---------------------|
| Cauliflower leaves | Extraction medium | 50 | 2.79 ^{***} |
| | Error | 102 | 0.004 |
| Cabbage leaves | Extraction medium | 50 | 2.48 ^{***} |
| | Error | 102 | 0.013 |
| Turnip leaves | Extraction medium | 50 | 2.63 ^{***} |
| | Error | 102 | 0.004 |

Table 5. Color shade of non mordanted and mordanted (chemical & bio) cotton fabrics dyed with vegetable residual material.

| Plant dye source | Extracted dye shade | Chemical mordanted fabrics | | Bio mordanted fabrics | |
|------------------|---------------------|----------------------------|----------------|-----------------------|----------------|
| | | Pre mordanted | Post mordanted | Pre mordanted | Post mordanted |
| Cabbage | Light brown | Light brown | Brown | Yellowish green | Yellow |
| Cauliflower | Creamy white | brown | Light green | Yellow green | Dark yellow |
| Turnip | Olive green | Dark brown | dark brown | Yellowish green | Dark green |

Table 6. Qualitative determination of phytochemicals from three plants of Brassicacea.

| Plant name | Alkaloids | Tannins | Flavonoids | Saponins | Glycosides | Fats | Sugars | Carbohydrates | Phenolics | Terpenoids |
|-------------|-----------|---------|------------|----------|------------|------|--------|---------------|-----------|------------|
| Cabbage | + | - | + | + | + | - | + | + | - | - |
| Cauliflower | + | + | + | + | + | - | + | + | - | - |
| Turnip | + | + | + | + | - | - | + | + | - | - |

Table 7. Mean square values from ANOVA for mordanting of colorant material extracted from residual material of selected plants.

| Plant | Mordanting type | Medium | Degree of freedom | Mean square value | |
|----------------|--------------------|---------------|-------------------|-------------------|---------|
| Cabbage leaves | Chemical pre | Mordant | 23 | 3.18*** | |
| | | Error | 48 | 0.076 | |
| | Chemical post | Mordant | 23 | 5.89*** | |
| | | Error | 48 | 0.015 | |
| | Bio pre | Mordant | 29 | 16.95*** | |
| | | Error | 60 | 0.015 | |
| | Bio post | Mordant | 29 | 13.28*** | |
| | | Error | 60 | 0.013 | |
| | Cauliflower leaves | Chemical pre | Mordant | 23 | 2.03*** |
| | | | Error | 48 | 0.006 |
| | | Chemical post | Mordant | 23 | 3.22*** |
| | | | Error | 48 | 0.006 |
| Bio pre | | Mordant | 29 | 3.48*** | |
| | | Error | 60 | 0.005 | |
| Bio post | | Mordant | 29 | 11.49*** | |
| | | Error | 60 | 0.005 | |
| Turnip leaves | | Chemical pre | Mordant | 23 | 2.78*** |
| | | | Error | 48 | 0.235 |
| | | Chemical post | Mordant | 23 | 7.39*** |
| | | | Error | 48 | 0.075 |
| | Bio pre | Mordant | 29 | 6.72*** | |
| | | Error | 60 | 0.076 | |
| | Bio post | Mordant | 29 | 28.09*** | |
| | | Error | 60 | 0.078 | |

Table 8. Color fastness characteristics of chemical and bio mordanted fabrics using dye extract of vegetable residual material.

| Plant species | Mordanted fabrics | LF | WF | DRF | WRF |
|---|-------------------------|-----|-----|-----|-----|
| (Cabbage) <i>Brassica oleracea</i> L. var. <i>capitata</i> | Chemical pre mordanted | 3 | 3/4 | 4/5 | 4 |
| | Chemical post mordanted | 4 | 4/5 | 4 | 3 |
| | Bio pre mordanted | 3 | 3/4 | 4 | 4 |
| | Bio post mordanted | 4/5 | 3/4 | 4 | 3 |
| (Cauliflower) <i>Brassica oleraceae</i> L. var. <i>botrytis</i> | Chemical pre mordanted | 4 | 3/4 | 4 | 4/5 |
| | Chemical post mordanted | 3 | 3/4 | 4 | 4/5 |
| | Bio pre mordanted | 3/4 | 3 | 4 | 4/5 |
| | Bio post mordanted | 3/4 | 4 | 4/5 | 4 |
| (Turnip) <i>Brassica rapa</i> L. var. purple top | Chemical pre mordanted | 4 | 3 | 3/4 | 4 |
| | Chemical post mordanted | 4 | 3 | 3/4 | 4 |
| | Bio pre mordanted | 3 | 3/4 | 4 | 3 |
| | Bio post mordanted | 4/5 | 3 | 4/5 | 4 |

DRF= Dry rubbing fastness, **LF** = Light fastness, **WF** = washing fastness, **WRF** = Wet rubbing fastness

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