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### Original Research Article

## A potential benefit of brown seaweed (*Stoechospermum marginatum*) using for sustainable fabric dyeing

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Sustainable dyeing method

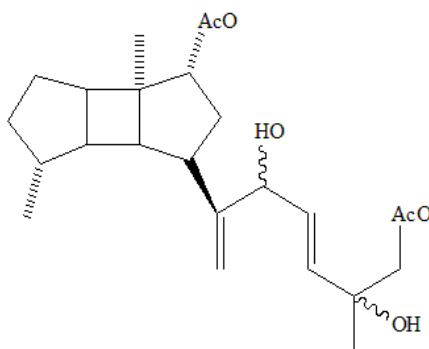
### ABSTRACT

Coastal areas are famous for its diversity and abundance of marine flora and fauna. Several tonnes of seaweeds grows annually and during low tide they are easily available either in growing or drift form. The current investigation was focus to target these marine flora to provide an alternative to toxic colorants. The brown seaweed, *Stoechospermum marginatum* were collected from the coastal area of Arabian sea during low tide. Seaweed colorants can be used as sustainable and eco-friendly source to make a value added product. Extraction process were optimized by varying solvent medium (alkali, acidic and distilled water), solvent concentration and extraction time. Eco-friendly metal salt like ferrous and aluminium sulphate were used to developed twenty-four soft shades on S/J 100% cotton. Dye exhaustion, color measurements and fastness properties such as wash, crock and light fastness of resultant fabric have been studied. The optimized condition of extraction process was 5g/L sodium hydroxide for 3hrs processing time. The dyed fabric having 5g/L sodium hydroxide extracted dye and mordanted with ferrous sulphate shows maximum exhaustion percentage as well as good to very good fastness property while the  $\Delta a$  and  $\Delta b$  values of color measurements shows that the dyed fabric has reddish-yellow shade.

### Introduction

Nature has dispersed colors throughout the universe and the human eye can recognize different colors and the variations in their shades. People used colors from the history of mankind for painting and drawing in the cave, women used for making themselves more beautiful and

attractive, dyeing and color the textile like carpets, rugs and clothes and leather materials by using roots, stems, barks, leaves and flower of various plants (Yusuf et al. 2017). Searching of colors or hues from indigenous sources has always gathered our interest due to their non-toxicity, renewability and biodegradability. Usually, the hues or colors of natural dyes and pigments have been found in red, yellow, orange, peach, green, blue and black and some types it can be classified on the basis of chemical constituents (Dyer, 1976; Ferreira et al. 2004; Samanta and Konar, 2011; Shams-Nateri et al. 2014). The natural colors are environmentally friendly and can be used on all types of natural fibers as well as on synthetic fibers (Elnagar et al. 2014). Due to the huge demand of colorants, a great deal of dyes has been synthesized for different industries i.e. food, textile, leather, electronics, plastic, beverages etc. Natural dyes can develop very rare and soft shades on different matrices as compared to synthetic dyes. However, the use of synthetic dyes may release some toxic chemicals into the environment during their processing, production or in finished products (Shahid et al. 2013). A large number of plants, animals and insects have been used for the extraction of color (Borges et al. 2012; Guinot et al. 2006; Sarkar and Seal, 2003) and their diversified use in textile dyeing (Samanta and Agarwal, 2009), functional finishing (Gupta et al. 2005), food coloration (Delgado-Vargas et al. 2000), cosmetics (Dweck, 2002), dye-sensitized solar cells (Hao et al. 2006), histological staining (Tousson and Al-Behbehani, 2011), as pH indicators (Mishra et al. 2012) and several other applications (Zheng et al. 2011) and disciplines (Kuswandi et al. 2012). Arabian sea are famous for the collection of various Seaweeds and Algae for research analysis (Ali et al. 2004). Researchers have isolated many known and new chemical constituents from various algae which exhibited cytotoxic, antibacterial and antiviral activities (Ali et al. 2003). Seaweeds are widely used as food products, fertilizers, fodder, pharmaceutical and in phytochemical investigations (Kilinç et al. 2013). The brown seaweed *Stoechospermum marginatum* (C. Agardh) Kützinger is belongs to phylum Phaeophycota of class Dictyotaceae. Chemical constituents of *S. marginatum* contain spatane derivatives which has both antimicrobial and anti-cancer activity (Chinnababu et al. 2015) (Figure 1).



**Figure 1.** Chemical structure of spatane derivatives.

Despite the search for non-toxic and eco-friendly color or hue from the indigenous or aquatic sources on textile, seaweeds have been rarely used as dye for the coloration of textiles (Janarthanan and Kumar, 2017). In this research, *S. marginatum* has been utilized as a natural colorants.

## Materials and Methods

### Materials

The brown seaweed *Stoechospermum marginatum* were collected during low tide in April, 2017 from Arabian sea. The collected sample was washed thoroughly with plenty of tap water to remove the sea salts, sand particles, animal casting and epiphytes attached on it. The washed sample was dried under shade for 2 days. The dried material of seaweed were grind into powdered form and stored at room tempertaure (Figure 2). An entire thalli of specimen were mounted on herbarium sheet and saved as voucher specimen in herbarium deapertment of Muhammad Afzal Hussain qadri biological research centre.



(a)



(b)

**Figure 2.** *Stoechospermum marginatum* (a) growing and (b) powdered form.

The Fabric Silk/Jersey, 100% Cotton, knitted at **Vanguard Circular** Knitting Machine using 100% cotton yarn 20/1, stitch length 0.135/cm, dia. 20", guage 22, on the machine GSM 170, finish GSM 205, needles 1380, RPM 36, wales per inch 32, course per inch 42 was obtained from Textile Mill for the use of mordanting and dyeing experiments. Fabric had been pretreated for experimental work and the Saclvos dyeing machine under controlled conditions by SEDOMAT 5000

used for further process. Sodium hydroxide (99%) from Sigma-Aldrich, Aluminum sulfate octadecahydrate (99.5%) from BDH, Ferrous Sulfate heptahydrate (99%) and acetic acid (99-100%) from Riedel-de Haën and laboratory grade distilled water were used during the course of the work.

### *Instruments*

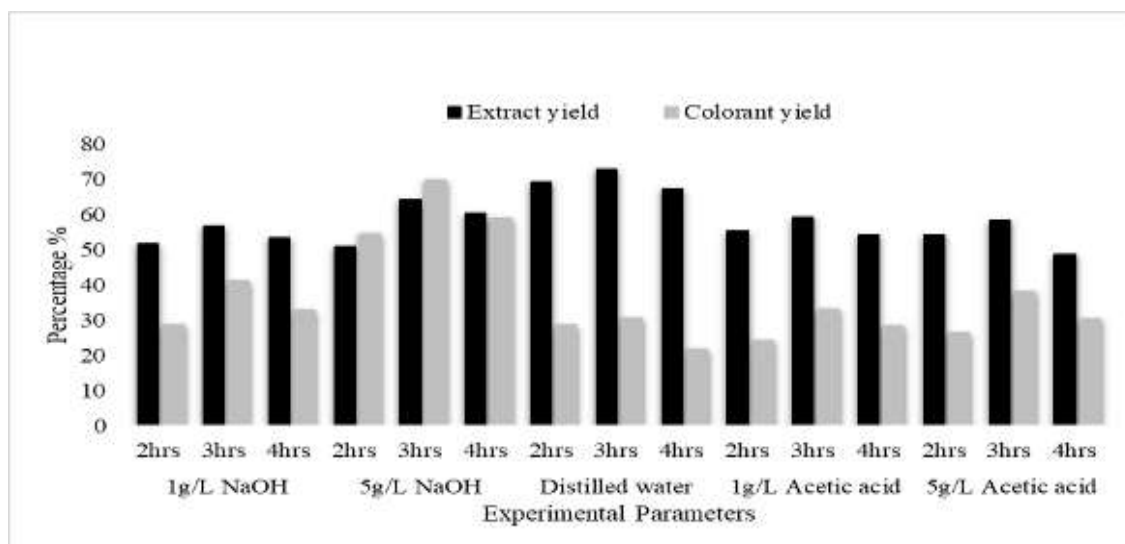
The extraction of dyes in different media were performed on Memmert 854 Schwabach water bath set at different temperature and time. The pH of dye bath solutions was measured using a pH (0-14) indicator strip by Merck KGaA, 64271 Darmstadt, Germany. Ultrasonication was employed for the equal distribution of dyes in the solution using Solid State/Ultrasonic T-14 B Sonicator. NICOLET evolution 100 spectrophotometer was used for determining the exhaustion rate of the dyed fabric before and after dyeing. Experimental trials such as temperature, extraction medium and extraction time were used to optimize the dye extraction process and to study the fastness properties and levelness on S/J 100% cotton fabric. Data Color F600 spectrophotometer (illuminated D65/10° observer) with Color tool software version 2.0.4 was used to evaluate CIE  $\Delta L$ ,  $\Delta a$ ,  $\Delta b$ ,  $\Delta E$ ,  $\Delta C$ ,  $\Delta H$ ,  $\Delta E_{cmc}$ , CIE whiteness and K/S. Each value was averaged from three samples.

### *Optimization of extraction condition*

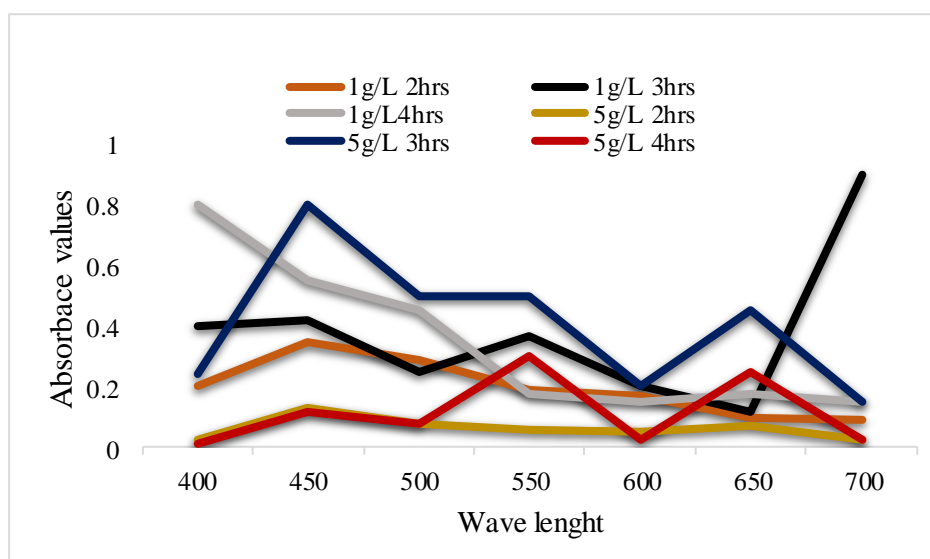
To optimize the extraction process, different trials have been performed. 10g of *Stoechospermum marginatum* was taken, powdered and used with distilled water, acetic acid and sodium hydroxide. Sodium hydroxide and acetic acid solutions were used as 1 g/L and 5 g/L having material-to-liquor ratio of 1:20 and heated at temperature of 80°C for 2, 3 and 4 hours. The mouth of flask was closed with aluminium foil to prevent excess evaporation of the solution. The extracted solution was then allowed to set at room temperature and filtered through clean cotton piece. The extracted dye solutions were dark brown in color. The absorbance values of all the extracted solutions were also recorded at the range of UV-VIS spectrum (400-700nm). The dye extracted in alkali medium shows highest absorbance values and are shown in Figure 4. All the extracts were then set for drying till a gummy extract was obtained. The extract yield and colorant percentage were also calculated by the following formula (Venkatasubramanian et al. 2009) (Figure 3),

$$\text{Extract yield \%} = \frac{\text{Total extract obtained (G)}}{\text{Amount of seaweed + Solvent used}} \times 100$$

$$\text{Colorant yield \%} = \frac{\text{Crude dye obtained (G)}}{\text{Amount of seaweed used}} \times 100$$



**Figure 3.** Total extract and colorant yield % at 80°C for 2, 3 and 4hrs extraction time by using sodium hydroxide, distilled water and acetic acid.

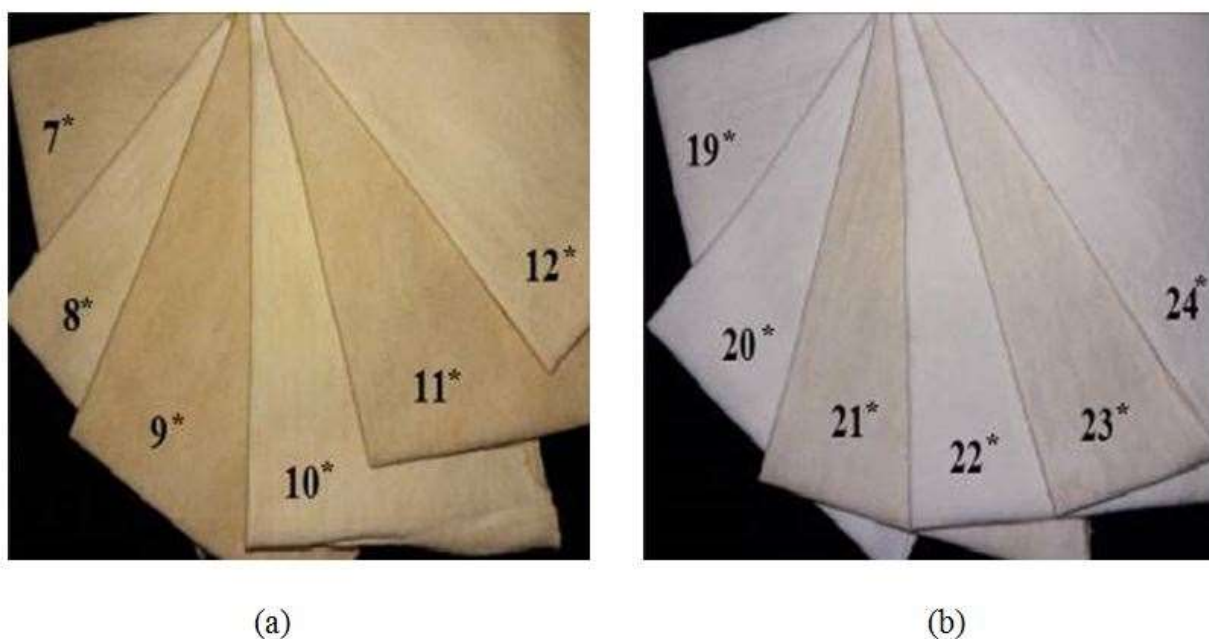


**Figure 4.** UV-VIS (400-700nm) values of extracts at 80°C for 2, 3 and 4hrs extraction time by varying sodium hydroxide concentration

#### *Procedure for mordanting and dyeing*

The extract which gives higher color strength was further utilized to optimize the dyeing condition. In pre-mordanting technique, fabric first mordanted with 20% on weight of fabric (owf) mordant solution having material-to-liquor ratio of 1:20 at 80°C for 30 mins. After mordanting, the sample was rinsed with cold water and air dried. Mordanted fabric was added to a dye bath containing 20% owf dye (crude dye), with a 1:20 material-to-liquor ratio at 80°C for 1 hr at pH 6.0 (acetic acid or sodium hydroxide were used as a fixing agent). Similar process was followed for post-

mordant method except the fabric first treated with dye bath solution than mordant. Figure 5 shows the significant results treated with ferrous and aluminum sulphate using 5g/L NaOH solution extracted dye by varying extraction time to 2, 3 and 4hrs.



**Figure 5.** Fabric treated with ferrous (a) and aluminum sulphate (b) using 5g/L NaOH dye extracted at 2, 3 and 4hrs.

#### *Dye exhaustion percentage*

The dye exhaustion rate were determined by measuring the absorbance values before and after dyeing the fabric. The absorbance value of all the samples were recorded in the range of 200-400nm. The dye exhaustion percentage were calculated by the following formula.

$$\text{Extract yield \%} = \frac{A_0 - A_1}{A_0} \times 100$$

Where A0 and A1 are the absorbance value before and after dyeing the fabric, respectively.

#### *Color strenght (K/S) and measurement*

Color strenght of the sample were measured by Data Color F600 spectrophotometer (illuminated D65/10° observer) with Color tool software version 2.0.4. The color strenght of all dyed fabric were recorded against 450 nm. The extract of *stoechospermum marginatum* shows maximum absorbance value at this wavelenght (Kundal et al. 2016).

The color measurement of all samples were measured in the visible range (400-700 nm) before and after dyeing using a Data color SF650 spectrophotometer having software version 2.0.4 and

calibrated on setting SCI UV 400. These reflectance spectra are used to deduce the color parameters with the assistance of CIE Lab ( $\Delta L$ ,  $\Delta a$ ,  $\Delta b$ ,  $\Delta E$ ,  $\Delta C$ ,  $\Delta H$ ,  $\Delta E_{cmc}$  and whiteness).

Fastness testing and color measurements. A number of fastness tests like wash, crock and light were performed on the dyed fabric samples. The wash fastness of the dyed fabrics was measured through AATCC Test Method 61-2 on ATLAS Laundry-o-meter at 49 degrees for 30 min and 40 RPM. Dry and wet crock fastness was evaluated using the AATCC Test Method 8 on ATLAS Crock meter CM5. The change in color was assigned using the AATCC Chromatic Transference Scale. Light fastness was determined according to AATCC 16 E-2014 Test Method on SUNTEST+, SDL-ATLAS brand, Black penal temperature  $63 \pm 1$  °C, Chamber air  $43 \pm 2$  °C and Humidity  $30 \pm 5\%$ .

## Results and Discussion

### *Effect of Extraction Parameters on Dye Yield*

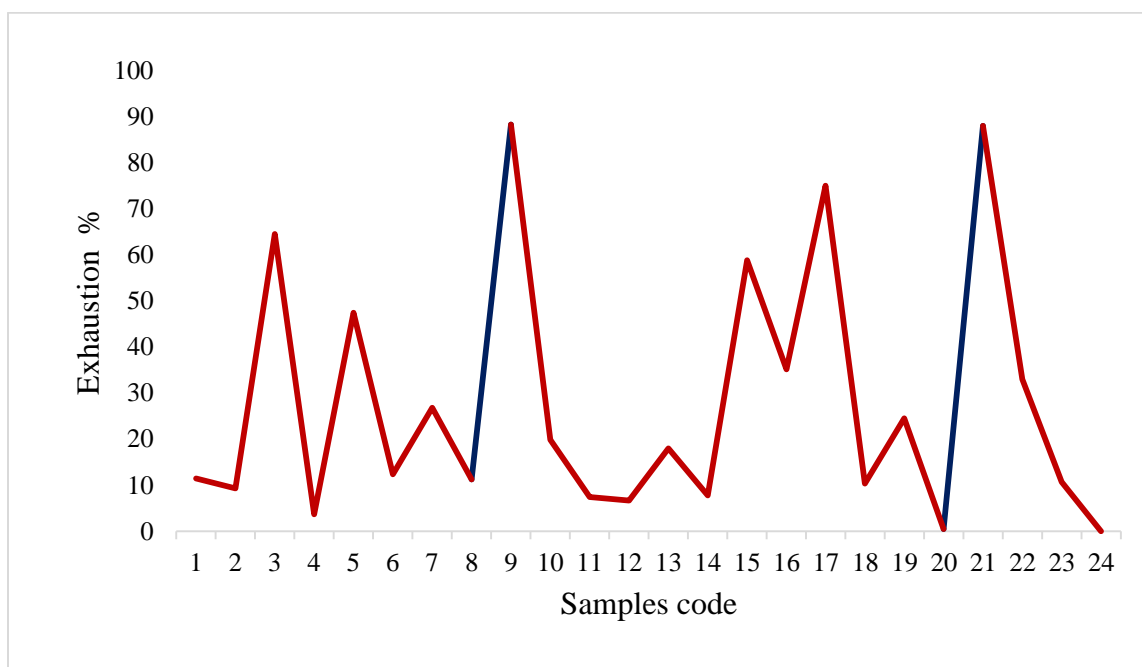
The extraction of natural colorants from *Stoechospermum marginatum* studies revealed that sodium hydroxide were more effective than acetic acid and distilled water. The alkali medium enhance the extraction process by rupturing the hydrocolloide cell wall material of brown seaweed and providing the greater number of OH ions for color solubility (Ali, 2007). Different concentration of NaOH solution gives high colorant strenght and crude dye yield. The effectiveness of extraction media can be expressed as 5g/L NaOH>1g/L NaOH>5g/L acetic acid>1g/L acetic acid>distilled water (Riffat et al. 2019). Extraction time has significant effect on extraction process by increasing time the cell wall of material were softened and released highest extract by weight. Colorant percentage of the extract were good at 2hrs processing time, as the time increase to 3hrs colorant percentage were maximum while it is decreased with further increasing time to 4hrs because the extract obtained its saturation level at 3hrs processing time after that it is further decline. This may be due to decomposotion of coloring component at longer extracting time (Nagia and El-Mohamedy, 2007). The optimized condition for dye extraction is 5g/L sodium hyrdoxide solution at 80°C for 3hrs extraction time.

### *Dye Exhaustion Percentage*

The percentage of the dye molecules bond to fabric is called as exhaustion rate or dye uptake. The exhaustion % of the sample were observe maximum in samples dyed with three hours extracted dye after that it is gradually decline. Dye bath temperature plays an important role in the exhaustion % because the dye molecule diffuse more rapidly with fibers which also increases the dye uptake. Similarly as we increase the NaOH concentration the exhaustion percentage increases



because NaOH it self act as a fixing agent which fix the dye molecule to the substrate. Pre-mordanting with ferrous sulphate has maximum dye uptake because the metal dye bonding were more stronger than aluminum sulphate (Figure 6).



**Figure 6.** Exhaustion percentage of sample dyed and with ferrous and aluminum sulphate.

#### *Effect of mordanting on dyeing and color strength (K/S).*

The K/S values of dyed fabrics were lie between 0.0-0.19 and recorded against wavelength 450 nm. Mordanting with ferrous sulphate gave highest color strength as compared to aluminum sulphate. The metal and dye complex were stronger with ferrous sulphate due to strong chelating power while aluminum sulphate forms weak complex with the dye molecule. Color strength of the dyed fabric were maximum at three hours extraction time while it is slightly decrease at four hours extraction time because the dye molecules reached their saturation level at three after that it is gradually decline. Pre-mordanting techniques gave darker shade as compared to post-mordanting.

#### *Fastness properties and color measurement of dyed fabric.*

Wash fastness of dyed fabric were tested by staining on adjacent test sample acetate, cotton, nylon, polyester, acrylic and wool fabric has been found to be good to very good rating on gray scale. The dry crock fastness rating of the sample have good to very good rating while wet crock fastness rating were fair to excellent. Light fastness of samples also have good to very good rating (Table 1).



**Table 1.** Fastness properties of dyed fabric mordanting with ferrous sulphate and aluminum sulphate.

Samples code	Samples Abb.	Acetate	Cotton	Naylon	Poly- Ester	Acrylic	Wool	Dry	Wet	Light Fastness
		Before Curing - 2A Wash Fastness – Staining					Crocking			
1	FO21	4.5	4	4.5	4.5	4.5	4.5	4.5	3.5	4
2	FO22	4.5	4	4.5	4.5	4.5	4.5	4	3.5	4
3	FO31	4.5	4	4.5	4.5	4.5	4.5	4.5	4.5	4
4	FO32	4.5	4.5	4.5	4.5	4.5	4.5	4	5	4
5	FO41	4.5	4	4.5	4.5	4.5	4.5	4.5	3.5	4
6	FO42	4.5	4	4.5	4.5	4.5	4.5	4	3.5	4
7*	Ff21	4.5	4	4.5	4.5	4.5	4.5	4	3.5	4.5
8*	Ff22	4.5	4	4.5	4.5	4.5	4.5	5	4	4
9*	Ff31	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4
10*	Ff32	4.5	4	4.5	4.5	4.5	4.5	4.5	4	4
11*	Ff41	4.5	4	4.5	4.5	4.5	4.5	4	3	4
12*	Ff42	4.5	3.5	4.5	4.5	4.5	4.5	4	3.5	4
13	AO21	4.5	4.5	4	4.5	4.5	4.5	5	4.5	4
14	AO22	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4
15	AO31	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4.5
16	AO32	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4
17	AO41	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4.5
18	AO42	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4.5
19*	Af21	4.5	4.5	4.5	4.5	4.5	4.5	5	5	4
20*	Af22	4.5	4.5	4.5	4.5	4.5	4.5	5	5	4.5
21*	Af31	4.5	4.5	4.5	4.5	4.5	4.5	5	5	4
22*	Af32	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4
23*	Af41	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4.5
24*	Af42	4.5	4.5	4.5	4.5	4.5	4.5	5	4.5	4.5

Notes. F = ferrous sulphate, A = aluminum sulphate; O = 1g/L NaOH, f = 5g/L NaOH; 2, 3 and 4 = 2hrs, 3 hrs and 4 hrs; 1 = Pre-Mordant, 2 = Post-Mordant.

The color differences of dyed fabrics are calculated as sample and standard values. All the pre and post-mordant samples shows lowest lightness values which show that treated fabric has darker shades. Shade depth was darker in case of ferrous sulphate than aluminum sulphate. The  $\Delta a$  and  $\Delta b$  values are chromaticity coordinate and shows direction towards and away from the color sphere. The negative a values represent green or positive a represent red. The negative b values shows blue and positive b shows yellow. Sample mordanted with ferrous sulphate shows +a values which shows shade are red than green. Aluminum sulphate fabrics has -a values shows shade are green than red. The b values of all dyed fabric are positive which shows samples are yellow than blue. Total Color Difference ( $\Delta E$ ) values are based on the values of L, a and b. The mordant ferrous sulphate showed more color difference as compared to aluminum sulphate.  $\Delta C$  values are all

positive and the fabrics that were treated with ferrous sulphate gave higher  $\Delta C$  values as compared to aluminum treated samples.

**Table 2.** Color measurement of samples dyed with ferrous sulphate and aluminum sulphate.

Samples code	CIE $\Delta L$	CIE $\Delta a$	CIE $\Delta b$	CIE $\Delta E$	CIE $\Delta H$	CIE $\Delta C$	$\Delta E_{cmc}$	CIE Whiteness	K/S Values
1	-13.37	5.63	23.49	27.61	-1.94	24.08	30.19	-23.37	0.1605
2	-14.03	6.91	23.69	28.39	-2.34	24.57	30.86	-26.4	0.1443
3	-13.14	5.61	21.83	26.09	-1.98	22.45	28.2	-22.46	0.1852
4	-15.45	7.74	27.12	32.16	-2.47	28.1	35.24	-30.39	0.1456
5	-13.95	5.7	24.89	29.1	-1.92	25.46	31.9	-24.32	0.1533
6	-12.68	5.93	21.01	25.24	-2.12	21.72	27.31	-22.65	0.1373
7*	-13.11	3.97	22.18	26.07	-1.43	22.49	28.19	-19.11	0.1381
8*	-10.53	2.73	17.12	20.29	-1.11	17.3	21.71	-13.8	0.1029
9*	-16.86	2.96	20.99	24.79	-1.12	21.16	26.54	-16.37	0.1988
10*	-13.09	5.01	26.21	30.18	-1.67	26.64	33.31	-23.47	0.1554
11*	-13.5	6.85	30.16	35.05	-2.11	30.86	38.61	-30.22	0.1333
12*	-12.29	4.56	24.24	28.51	-1.57	24.62	30.85	-21.59	0.1035
13	-4.53	-0.1	5.77	7.33	0.01	5.77	7.29	-3.14	0.0380
14	-3.26	-0.62	6.12	6.96	0.3	6.15	7.68	-2.31	0.0306
15	-4.22	-0.04	6.32	7.6	-0.03	6.32	7.93	-3.45	0.0352
16	-2.65	0.25	5.08	5.74	-0.19	5.08	6.35	-3.44	0.0269
17	-3.33	0.04	4.95	5.97	-0.07	4.95	6.22	-3.03	0.0295
18	-2.33	-0.37	4.21	4.83	0.19	4.22	5.28	-2	0.0251
19*	-5.19	-1.23	7.95	9.57	0.57	8.02	10.09	-1.95	0.0411
20*	-3.7	-1.45	6.22	7.38	0.76	6.34	7.99	-0.86	0.0333
21*	-6.55	-1.16	8.23	10.59	0.52	8.3	10.51	-2.21	0.0527
22*	-4.19	-1.28	6.59	7.91	0.64	6.68	8.42	-1.32	0.0395
23*	-5.77	-1.07	8.64	10.44	0.47	8.69	10.93	-2.51	0.0494
24*	-3.62	-0.59	6.57	7.52	0.27	6.59	8.23	-2.54	0.0333

The positive  $\Delta C$  values show that all the samples are more saturated than the standard.  $\Delta H$  values show the magnitude of change in hue. Fabrics treated with ferrous sulphate showed negative change in  $\Delta H$  whereas those treated with aluminum sulphate showed positive. The hue values of all sample mordanted with ferrous sulphate were lie between 0.54 to -2.11 while aluminum sulphate samples values were lie in the range of 0.01 to 0.76.  $\Delta E_{cmc}$  defines an elliptical color difference; all ferrous sulphate treated fabrics for pre and post-mordant showed higher elliptical color difference as compared to the aluminum sulphate. CIE Whiteness values were all negative (red hue or less

blue) except that the values for ferrous sulphate were on higher negative as compared to aluminum sulphate (Table 2).

## Conclusion

The textile sector facing a critical problem regarding the environment and health care product based on the synthetic dyes. To provide the potential solution and to overcome these problem seaweed extract may use as ecofriendly colorants. Seaweeds or macro algae are commonly grow in coastal areas either in growing or drift form. They are the rich source of bioactive compound like chlorophyll, carotenoids, phycobiliproteins and sulphated polysaccharides, these are used for extraction of natural dyes in textile or other areas of study. The present research focused on the extraction condition and application of natural colorant from *Stoechospermum marginatum* on S/J 100% cotton fabric. Shades have been developed by varying experimental parameters and mordants. Mordanting with Ferrous sulphate produce darker shade as well as maximum exhaustion percentage and K/S values as compared to aluminum sulphate. Extraction time has considerable effect on extraction process while slight effect on the shade development. Fastness rating of maximum fabrics were lie 4.5 to 5, the seaweed dyed fabric is acceptable after standard testing method.

## Conflicts of Interest

No conflicts of interest have been declared.

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