



Extraction of Natural Dye from the Root of *Rumex Abyssinicus* for Textile Dyeing Application

Melkamu Birlie Genet^(✉), Asres Mekonnen Admas, Animaw Gerem Abeje, and Felegush Enyew Melese

Bahir Dar Institute of Technology, Bahir Dar University, Bahir Dar, Ethiopia

Abstract. Dyes extracted from natural sources have emerged as important substitutes to synthetic dyes. In this study, an environmental friendly natural colorant was extracted from the root of *Rumex abyssinicus* (Mekmeko) by using an aqueous extraction method. The effect of temperature and pH on the extraction of natural dye was studied and the best condition for dye extraction was obtained at pH 10 and temperature of 80 °C. UV-VIS absorption spectrum indicates a λ_{max} peak at 280 nm and this value is associated to anthraquinone dyes. Structural characterization by FT-IR revealed the presence of chromophores compounds like C=O, OH, -C=N- and C=C of aromatic. The coloring potential of the extracted dyes has been studied using selected mordant types, pH, and dyeing techniques. The simultaneous mordanting method at acidic media and iron sulfate mordant type shows very good (4) and very good to excellent (4–5) grade colorfastness characteristics to washing and rubbing.

Keywords: *Rumex Abyssinicus* · Natural dye · Mordant · Cotton fabric

1 Introduction

Dyeing with natural colorants was one of the oldest techniques practiced since the prehistoric period. Natural dyes extracted from plants, minerals, and insects were used to stain hides, decorate shells, feathers, paint the ancient story on the walls of caves, color food substrate, color natural protein fibers like wool, silk and cotton [1]. Primitive men also used plant dyestuffs to color animal skin and dye their own skin during religious, traditional festivals and wars. Dyes might have found unintentionally, but their advantage has become extremely a part of man's habit that it is hard to think of a civilized world without colorants [2, 3].

Until the end of the 19th century, natural dyes were the major colorants for textile industries. The discovery of first synthetic dye mauveine, a basic dye type, by Sir W. H. Perkin in 1856 led to almost a complete replacement of natural dyes in textile industries [4, 5]. The rapid decline of the use of natural dyes after the introduction of synthetic dyes was due to its drawbacks like the inconsistency of colors, less availability, lack of fastness, etc. [6]. Synthetic dyes are produced from cheap petroleum raw materials

and these dyes are become popular due to its simple production, variety of colors, reproducible application processes, and the consumer's demand for quality products having superior fastness properties at a low price [5, 7]. Currently, there is excessive use of synthetic dyes in numerous industries estimated around 10,000,000 tonnes and approximately 10,000 different types of dyes and pigments are manufactured world-wide annually [8, 9]. The tremendous production rate and use of synthetic dyes in various industries led to the release of a vast amount of colored wastes into the environment. This poses a series health and ecological problem to the world since synthetic dyes are hazardous, toxic and carcinogenic, non-biodegradable, and non-renewable in nature. As a result, its application is slumped tremendously in textile and other dyeing industries in recent times [10]. Textile industries alone uses huge amount of water during the dyeing of fabrics. The effluents released from these factories contain a heavy load of hazardous chemicals including dyes used during textile processing significantly pollutes the natural ecosystem. It is estimated that 10–25% of textile dyes are lost during the dyeing processes [7] and 2–20% of such dyes are directly discharged as aqueous effluents into the environment [11]. It is difficult to remove such effluents that affect aquatic biota [12], rivers, soils, crops, etc. by conventional water treatment methods [6]. Recently, the interest of using natural dyes in textile industries has been growing rapidly due to the stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes. Some countries like Germany, USA, India, and Netherland imposed a ban on the use of some synthetic dyes [13–15].

Natural dyes constitute colorants that are obtained from flora, fauna, and minerals without any chemical processing has gained momentum due to increased demand for these dyes by the food, pharmaceutical [16], cosmetic as well as the textile coloration industries [17]. Natural dyes are mostly eco-friendly, biodegradable, less toxic, less allergenic, and renewable [18] dyes. These features and a growing concern for the environment have created a niche market for the industrial-scale use of natural colorants [19, 20]. Nature has gifted us with more than 500 dye-yielding plant species, and all colors of the rainbow are obtained from plants. They can provide not only a rich and varied source of dyestuff, but also the possibility of an income through the sustainable sale of these colorant plants. It has been reported that natural dyes can be successfully extracted from various plant sources like henna leaves, Mahogany, Ketapang, Tamarind, Mangosteen, Mango, Guava, papaya, Banana, and Onion, etc. [21, 22]. Natural colorant from different plant parts such as roots, leaves, barks, twigs, stems, heartwood, bark, wood shavings, flowers, fruits, rinds, hulls, husks, trunks can be extracted through different methods. Natural colorants can be extracted from its source by aqueous extraction, alkali or acid extraction, microwave and ultrasonic-assisted extraction, fermentation, enzymatic extraction, solvent extraction, and supercritical fluid extraction methods [23].

Utilizing natural dyes in textile industries has certain constraints like low dye uptake, low color yield, the complexity of dying process, reproducible results, limited shades and inadequate fastness properties which can be solved using chemicals called mordant [24, 25]. Mordants are metal salts that can escalate the binding efficiency of natural dye on cotton fabrics. Metal ions of mordants act as electron acceptors for electron donors to form coordination bonds with the dye molecule, making them insoluble in water. Copper sulfate, ferrous sulfate, chromium sulfate, stannous chloride, zinc sulfate, alum,

etc. are the commonly used mordants that make the fabric to have good colorfastness properties [26, 27]. Colorfastness is the resistance of a material to change any of its color characteristics or extent of transfer of its colorants to adjacent white materials in touch. Generally light fastness, wash fastness and rub fastness are considered for textile fibers.

Natural dyes can be classified based on colors, chemical constitution, application, and origin [28–30]. On the basis of color, natural dye can be classified as red, blue, yellow, green, black, brown, orange, and other derived dye types. Natural dyes have a complex chemical constitution. Unlike synthetic dyes, they are normally not a single entity but a mixture of closely related chemical compounds. On the basis of major chemical constituents present, they are divided into anthraquinone, indigoid, naphthoquinones, carotenoid, flavonoid, and Tannin-based dyes. Natural dyes are also classified based on the method of their application such as mordant dyes, direct dyes, vat dyes, acid dyes, and basic dyes, and disperse dye. There are three major sources from which natural dyes are extracted such as plants, minerals, and animals [31].

Ethiopia is rich in biodiversity and yet it doesn't use its natural resources effectively. Among its natural resources which are not utilized properly is the *Rumex Abyssinicus* (Mekmeko in local Amharic language) plant. *Rumex Abyssinicus* is widely spread throughout Ethiopia at altitudes between 1200 and 3300 m and some east African countries. This plant is native to Ethiopia (*Abyssinicus* means from Abyssinia, Ethiopia) [32]. *Rumex abyssinicus* is a member of the family of Polygonaceae and most of the species of Polygonaceae genus contain phytoconstituents, namely, flavonoid, anthraquinone, and triterpenoids. *Rumex abyssinicus* is a perennial herb, which grows up to 3 to 4 m, with the thick and fleshy rhizome. The root of *Rumex Abyssinicus* mainly consists of anthraquinone and flavonoid based colorant such as red and yellow dyes due to its rhizome [32, 33]. Traditionally Ethiopian Women used this plant root extensively to color their feet and hands during different religious festivals and other events to keep their beauty. This plant has also different antimicrobial activities and used as a cleaning agent to remove wastes. So, it is possible to extract natural dye from this plant for the textile dyeing application. There are not enough published research works on this indigenous plant for textile dyeing application and this study tries to fill the gap by providing some information about its dyeing performance in cotton fabrics.

This study aims to extract eco-friendly natural dye from the root of the *Rumex abyssinicus* plant for textile dyeing application using aqueous extraction method. The dye was extracted by varying pH and temperature at fixed extraction time (1 h). The coloring performance was studied by applying the extracted dye on the cotton fabrics at different dyeing conditions, pH, and mordants types.

2 Methodology

2.1 Raw Material Collection and Preparation

The roots of *Rumex Abyssinicus* used in this study were purchased from the market in Bahir Dar city, Amhara national regional state, Ethiopia. Then the root was washed by distilled water to remove mud and impurities. The cover of the cleaned root was peeled and cut into small pieces using a knife manually. The chipped root was dried on open-air by sunlight. Finally, the dried sample was crashed by disc mill and it was sieved to get a

uniform particle size passing through 0.5 mm sieve size. Figure 1 shows the root, peeled and powder of the *Rumex Abyssinicus* plant.

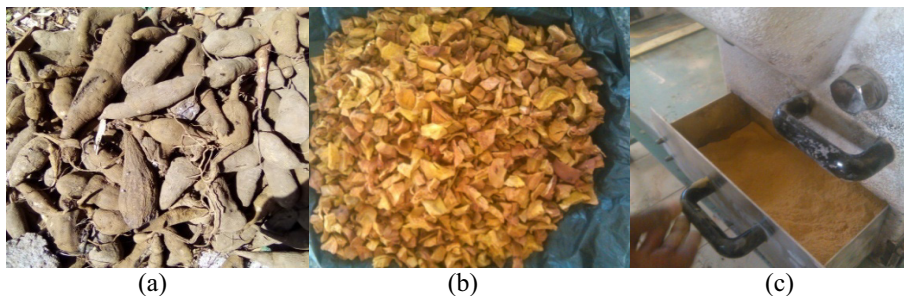


Fig. 1. *Rumex Abyssinicus*; root (a), chipped root (b) and powder (c)

2.2 Extraction of Dye

The aqueous dye extraction method is used to extract dye from the root of this plant due to its easy and simpler operation. Twenty-five grams of the *Rumex abyssinicus* root powder was added to the round bottom flasks containing 250 ml of distilled water (1:10 w/v) in acidic (pH-4), neutral (pH-7) and basic media (pH-10) at 40, 60 and 80 °C extraction temperatures at a constant extraction time of one hour. The pH of the acidic and basic medium was regulated by using 0.1M of H₂SO₄ and NaOH. The temperature of each sample was maintained by using a hot water bath. Then, the round bottom flask that contained the extracted dye solution was put on the mechanical shaker for two hours to effectively homogenize it. Finally, the solution was filtered by using a muslin cloth and the filtrate was used for dyeing.

2.3 Characterization of Dye

Identification of the Formation of Anthraquinone Dye: The dye extracted from the root of *Rumex abyssinicus* mainly contains anthraquinone colorant type [32, 33]. So, the formation of this colorant in the crud extract can be checked using either aqueous ammonia or sodium hydroxide solution. In this study, the presence of the anthraquinone colorant was checked by using sodium hydroxide solution. 3 ml of the crude dye extract from *Rumex abyssinicus* root powder was added to the test tube containing 1ml of 10% sodium hydroxide solution to confirm the presence of anthraquinone colorant type.

UV - Visible Spectroscopy: The wavelength of the maximum absorbance of an extract was determined by ultraviolet (UV-Vis) spectrophotometer (PerkinElmer, lambda 35). One milliliter of the extracted dye was diluted in 100 ml distilled water for UV analysis. The diluted dye extract was scanned from 200 to 700 nm to generate the characteristic absorption spectra of the sample and the corresponding peaks.

FTIR Spectroscopy: 2 mg of powder extract was mixed thoroughly with 200 mg of potassium bromide (KBr) and homogenized in an agate mortar. The mixture was then placed in the sample compartment of Infrared Fourier Transform Spectroscopy (FT/IR-6600typeA) to determine the main functional groups responsible for light absorption. The measurement was conducted between the ranges from 400 to 4000 cm^{-1} at a resolution of 4 cm^{-1} .

2.4 Dyeing Method

Scouring and Bleaching of Cotton Fabric: The desized cotton fabric samples obtained from Bahir Dar textile share company were washed in a solution containing 3% NaOH, 2.5% soda ash (Na_2CO_3), and 1% detergent at a temperature of 90 °C for 3 h. before dyeing of the cotton fabrics by keeping the material to liquor ratio 1:20. The scoured fabric was washed with tap water and it was dried at room temperature. The scoured fabrics were soaked by hydrogen peroxide for 30 min prior to dyeing or mordanting to bleach the fabric effectively [34].

Mordanting and Dying: Chemical mordants have been used to improve the fastness property of the natural dye. In this study, aluminum sulfate, iron sulfate, and copper sulfate mordants were used. Three different mordanting techniques were applied to dye the fabric such as pre mordanting, Simultaneous mordanting, and post mordanting conditions.

Pre-mordanting Method: In this method, the mordants were used to treat the fabrics before dyeing. Two grams of iron sulfate, aluminum sulfate and copper (II) sulfate mordants were dissolved in 500 ml distilled water separately. The scoured cotton samples were heated at a temperature of 60 °C for 50 min. The pre-mordanted cotton fabrics were allowed to dry without washing. After the mordanted fabrics were dried completely the fabrics were rinsed in the extracted dye to color it.

Simultaneous Mordanting Method: Two grams of iron sulfate, aluminum sulfate, and copper (II) sulfate mordants were added into extracted dye in a separate beaker. Then the scoured cotton fabrics were placed in the mixture and it was allowed to heat at a temperature of 60 °C for 50 min.

Post mordanting method: The dried scoured cotton fabric was immersed in extracted dye. Then, the dried and dyed cotton fabrics were treated with two grams of iron sulfate, aluminum sulfate, and copper (II) sulfate mordants separately in 500 ml distilled water and heated at a temperature of 60 °C for 50 min.

For all the mordanting techniques, the cotton fabrics were dyed directly with the dye extract by keeping the material to liquid ratio of 1:20. All the mordanting methods have done as described [35, 36]. The brine solution was added twice at a time interval of 15 min and finally, sodium carbonate was added for fixing purpose. Figure 2 below shows different dyed cotton fabric samples.



Fig. 2. Dyed cotton fabrics

2.5 Evaluation Method of Color Fastness of the Dyed Fabrics

Fastness to Washing: The washing fastness of the dyed cotton fabrics were conducted out using a Launder-O-meter by following the ISO standard Test No. 3 (ISO 105-C03:1989) procedures [37]. A 4 cm × 10 cm dyed cotton fabric was stitched around the edges of a white cotton sample. The dyed fabric was washed by a mixture of 150 ml water and 75 g of soap with 10 washing balls at a temperature of 60 °C for 35 min. After the treatment, the composite specimen was removed and rinsed in cold water and then dried in an oven. The dried sample was then assessed for change in color of dyed specimen and staining of adjacent fabric with grey scale and grading was given.

Fastness to Rubbing: Colorfastness dyed cotton fabrics to dry rubbing and wet rubbing fastness were tested as per AATCC 8–2007 test method using manually operated Crock meter and grey scale [37]. The specimen to be measured was rubbed against perfectly scoured and bleached fabric of dimension not less than 22 cm × 10 cm. For the determination of dry rub fastness, the sample to be tested was rubbed 10 times in 10 s in a dry condition, whereas for wet rub fastness determination, the same procedure has been adopted by wetting out the rubbing cloth and squeezing it to 100% expression. The staining on the rubbing fabric was assessed with the standard Grey Scale. Figure 3 depicts crock meter, lauder-o-meter, and grey scale.

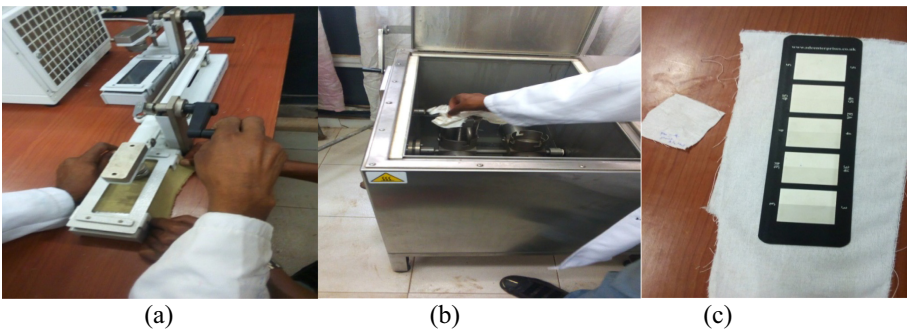


Fig. 3. Crock meter (a), Launder-o-meter (b) and Grey scale (c)

3 Result and Discussion

3.1 The Effect of pH and Temperature on Dye Extraction

Figure 4 shows the effect of extraction pH and temperature on the absorbance of the extracted dye. As the pH changes from acidic to neutral and neutral to basic medium, the absorbance of the extracted dye increased tremendously. The extracted dye at the basic media exhibits a maximum absorbance of 0.783 A. The reason for extracting more coloring components in alkaline solution was due to the presence of acidic phenolic groups in the root of *Rumex abyssinicus*, which reacted with alkali and form more soluble salts in water. The solubility of the coloring component increased due to an increase in ionization of hydroxyl groups in the alkaline medium. Similarly, the absorbance of the extracted dye increased when the extraction temperature rises from 40 °C to 80 °C. It is obvious that as the temperature increases the ruptures of the root of the *Rumex abyssinicus* cell wall increase enormously and as a result, more dye components were released into the extract. Higher color absorbance was observed at the extraction temperature of 80 °C. Higher extraction temperature can cause damage to protein-based fiber and so, the extraction temperature of 80 °C can be used as the optimum temperature in order to obtain greater color strength in the root extract.

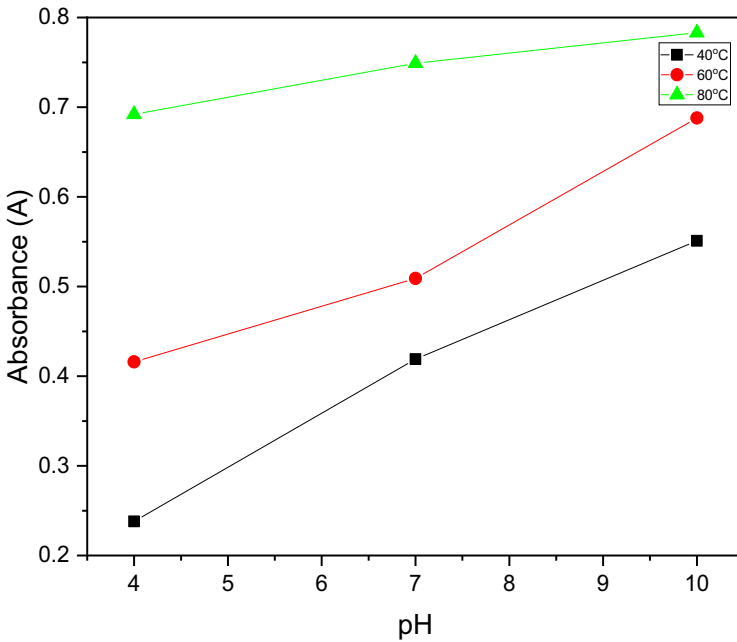


Fig. 4. Effect of pH and Temperature on dye extraction

3.2 Characterization of the Extracted Dye

Identification of Anthraquinone Colorant in Rumex Abyssinicus Root Extract:

Crude extracts of certain plants show color formation when treated with certain chemical compounds. As it is shown in Fig. 5, the formation of red color was observed when the aqueous extract was treated by NaOH. This result confirms the presence of hydroxyl anthraquinones, which is a major color bearing component in Rumex abyssinicus root extract [38]. Anthraquinones are generally yellow, orange, or brown colored pigments. Anthraquinone can be distinguished from benzoquinones and naphthoquinones as they usually give red solutions on a reduction in alkaline solution [39]. This implies that the anthraquinone colorant is the major constituent of the Rumex abyssinicus root extract.



Fig. 5. Anthraquinone colorant test

UV- Visible spectroscopy Analysis: Figure 6 shows the absorption spectrum of the aqueous dye extract from the root of the Rumex abyssinicus plant. A maximum absorbance peak (λ_{max}) was detected at 280 nm with an absorbance of 0.480977 Å in the UV visible region. Anthraquinones shows a strong quinonoid electro transfer at this absorption peak. The absorption band at λ_{max} 280 nm indicate characteristics absorption chromophore center for anthraquinone [33]. But the λ_{max} value obtained by other researchers does not match with those that compare to the anthraquinone derivatives like chrysophanol (λ_{max} = 440 nm), Emodin (λ_{max} = 445 nm) or Physcion (λ_{max} = 433 nm) in the invisible region [32]. This fact shows a bathochromic effect likely caused by co-pigmentation mechanisms. It is a consequence of a typical anthraquinone intermolecular association within flavonoids like tannins and catechins or glucocyclic groups, which can act like co-pigments.

FTIR Spectroscopy Result Analysis: The FT-IR spectrum of Rumex abyssinicus root powder extract showed the prominent peaks at 3393.1375, 2918.7346, 2363.3362, 1386.5677, 1324.8568, 1051.0145, 782.9576, and 620.9664 cm^{-1} as shown in Fig. 7. The broad peak appeared at 3393.1375 cm^{-1} assigned to O-H stretching vibrations. This band is caused by the presence of alcoholic and phenolic hydroxyl groups involved in hydrogen bonds [40, 41]. This verifies the presence of poly phenolic groups which are abundantly present in this plant. This polyphenolic compound helps in interlocking majority chromophore to get adhere to fabric and thus increase dye fastness. The

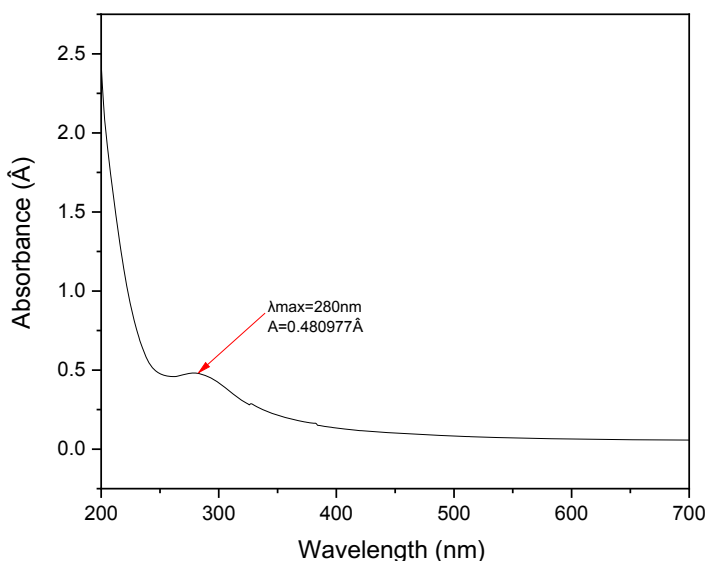


Fig. 6. UV-VIS spectra of Rumex abyssinicus root dye extract

bands at 2918.7346 cm^{-1} assigned to C-H stretching vibrations of the methoxyl group [42]. The peak at 2363 cm^{-1} confirms the presence of Nitriles, Azides, and alkynes. The stretching vibration at 1611 cm^{-1} confirms the presence of -C=O conjugates with the ring double bonds giving a -C=C-, -C=N-, and NH groups [42]. The peak at 1386 and 1324 cm^{-1} confirms the presence of nitro-compounds, alkanes and alkenes. The peak at 1051 cm^{-1} emanates from primary and secondary alcoholic groups [42]. The peak at 782 cm^{-1} shows the absorption band caused by deformation vibration of C-H bends on the benzene ring and it indicates the presence of aromatics [42–44]. The peak at 620 cm^{-1} confirms the presence of aromatic compounds. The presence of the main anthraquinone unit is confirmed by the carbonyl and hydroxyl vibrational absorption frequencies.

3.3 Colorfastness Properties of Dyed Cotton Fabrics

The colorfastness grade is equal to the grey-scale step which is judged to have the same color or contrast difference [45]. All the dyed cotton fabric samples recorded acceptable fastness grades except the non-mordanting fabrics. Table 1 shows the colorfastness properties of dyed cotton fabric under different dyeing conditions, mordant types, and pH. The mordanting methods, the mordant types and pH conditions efficiently improved to washing and rubbing colorfastness on cotton fabrics. The washing fastness (color change and staining) and rubbing fastness (dry and wet) grades of the cotton fabric recorded Good (3), good to very good (3–4), very Good (4) and very good to excellent (4–5) grades in colorfastness were recorded.

The dyeing of textile fabric at the pre-mordanting condition shows good, good to very good, very good, and very good to excellent grades of washing and rubbing fastness in

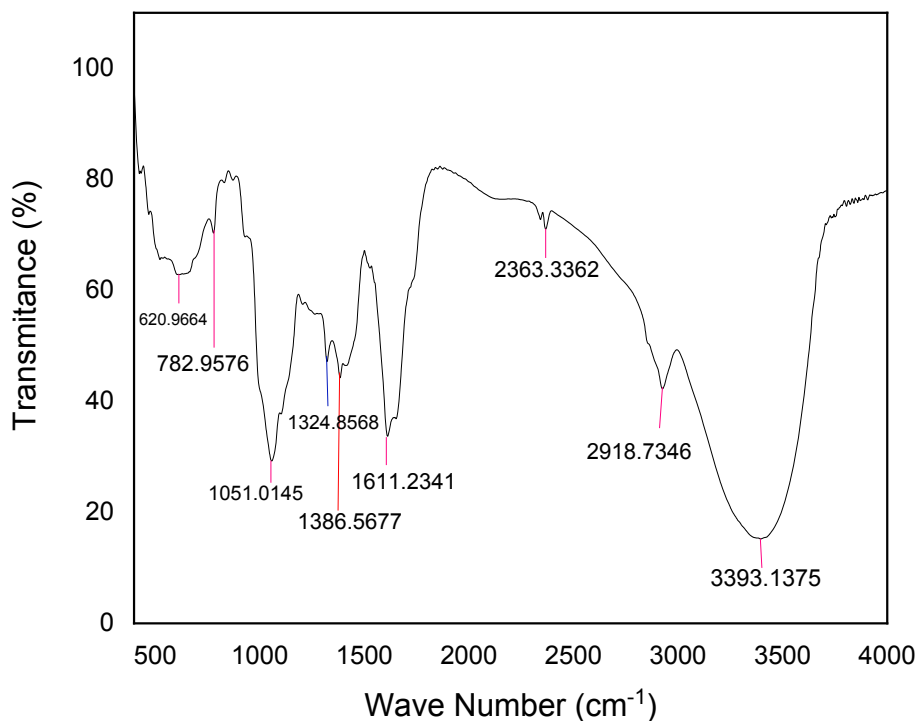


Fig. 7. FT-IR spectra of Rumex Abyssinicus root extract

aluminum sulfate, copper sulfate and iron sulfate with acidic, neutral, and basic media. The washing fastness of cotton fabric was recorded good (3), good to very good (3–4) and very good (4) grades in color changes and staining. Grades of rubbing fastness of the dyed cotton fabric both in wet and dry rubbing exhibit good (3), good to very good (3–4), very good (4) and very good to excellent (4–5) grades. Aluminum sulfate at pH 7 exhibits a very good (4) grade color fastness for both color change and staining washing fastness and very good to excellent (4–5) grade for dry rubbing and good to very good (3–4) for wet rubbing fastness respectively in the pre-mordanting conditions. In simultaneous mordanting condition, all mordant types and dyeing media show a good, good to very good, very good and very good to excellent grade colorfastness to washing and rubbing. Iron sulfate at pH 4 showed a greater colorfastness to washing and rubbing. Both the color change and staining washing fastness recorded a very good to excellent (4–5) colorfastness. Dry and wet rubbing color fastness of the cotton fabric exhibits a very good to excellent (4–5) and very good grade (4) colorfastness respectively. Similar to pre and simultaneous mordanting methods, post mordanting technique yields a good (3) good to very good (3–4), very good (4) and very good to excellent (4–5) grade colorfastness properties. The post-mordanting dyeing condition with iron sulfate at pH 10 shows improved colorfastness to washing and rubbing. Dyeing of cotton fabric by Rumex Abyssinicus extract without the application of mordant shows weak (2), weak to good (2–3) and good grade color fastness properties to washing and rubbing.

Table 1. Dying condition, mordant and pH effect on the performance of dyed cotton fabrics

Dying conditions	Mordant	pH	Fastness			
			Washing		Rubbing	
			Color Change	Staining	Dry	Wet
Pre-mordanting	Aluminum sulfate	4	3-4	4	4-5	3
		7	4	4	4-5	3-4
		10	4	3-4	4	4
	Iron sulfate	4	3	3-4	4	3
		7	3	3	4	3
		10	3-4	4	4	3-4
	Copper Sulfate	4	3	3-4	4	3
		7	3-4	4	4	3
		10	3-4	4	4	3-4
Simultaneous mordanting	Aluminum sulfate	4	4	4	4-5	3-4
		7	4	4	4-5	4
		10	4	3-4	4	3-4
	Iron sulfate	4	4-5	4-5	4-5	4
		7	4-5	4	4-5	4
		10	4	3-4	4	3-4
	Copper Sulfate	4	3-4	3	4	3
		7	4-5	4-5	4	3-4
		10	3-4	3	4	3
Post mordanting	Aluminum sulfate	4	3-4	3	4	3
		7	4	4-5	4-5	3-4
		10	3-4	4	4	4
	Iron sulfate	4	4	3-4	4	3-4
		7	4	3-4	4-5	4
		10	4-5	4	4-5	4
	Copper Sulfate	4	4	3-4	4-5	4
		7	3-4	4	4-5	4
		10	3-4	4	4	3-4
Non mordanting		4	2-3	2	3-4	2
		7	2	2-3	3	2
		10	3	2-3	3	2

Dyeing of the cotton fabric without mordants at acidic media gives good color fastness behaviors relative to that of the basic and neutral medium. It is difficult to use the *Rumex Abyssinicus* extract without mordant to dye the cotton fabric. Generally, the simultaneous mordanting method at acidic media and iron sulfate mordant type shows very good and very good to excellent grade colorfastness characteristics to washing and rubbing.

According to the result of this study, the simultaneous mordanting method is excellent dyeing technique, and it is recommended to dye the cotton fabrics in textile industries by the extracts of *Rumex abyssinicus* root powder. However, there was no significant difference in dry and wet rub fastness for the three mordanting methods. A remarkable improvement in colorfastness was recorded for all mordants and mordanting methods.

4 Conclusion

Natural dye was successfully extracted from the root of *Rumex abyssinicus* powder by the aqueous method. The best dyeing extraction condition was found at a basic medium (pH-10) and a temperature of 80 °C with an absorbance value of 0.783. The anthraquinone test of the extracted dye had red rose color and this confirms the presence of hydroxyl anthraquinone group in *Rumex Abyssinicus* root extract. A maximum absorbance peak (λ_{max}) was detected at 280 nm in the UV region and it indicates the *Rumex abyssinicus* plant contains anthraquinone colorant. The FTIR spectrum of the Mekmeko root powder contains O–H, C=O, C=C, C=N, and C-H functional groups, and this carbonyl, hydroxyl, and other aromatic compounds confirms the presence of the anthraquinone dye in the plant. The dye performance of *Rumex abyssinicus* root extract on cotton fabric tends to perform better with mordants compared to the non-mordanting conditions. Iron sulfate at pH 4 in the simultaneous mordanting technique showed a greater colorfastness to washing and rubbing. Both the color change and staining washing fastness was recorded a very good to excellent (4–5) colorfastness. Dry and wet rubbing color fastness of the cotton fabric exhibits a very good to excellent (4–5) and very good grade (4) colorfastness properties respectively. Generally, the extract of dye from the root of *Rumex abyssinicus* can be used as a promising source of natural colorant and a potential replacement for synthetic dye applicable for textile industries.

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