

## ***Nicotiana tabacum* as Natural Dye for Cotton- Influence of Gamma Irradiation and Mordants on Dyeing Performance**

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### **Abstract:**

**Background:** The present study explores the potential of *Nicotiana tabacum*, commercially grown in many countries for its leaves to be processed into tobacco as a renewable source of natural dye for cotton. The influence of gamma irradiation pretreatment and mordants on the dyeing properties of *Nicotiana tabacum* extract on cotton is studied.

**Materials and Methods:** The conditions viz., temperature, duration and pH of extraction of natural dye from the dried leaves of *Nicotiana tabacum* and the conditions for dyeing cotton fabric were optimized. The influence of gamma irradiation (1 to 5 kGy) on the colour strength, colour coordinates and colour fastness properties of the cotton fabrics dyed with *Nicotiana tabacum* was assessed. The irradiated samples were also pre-mordanted with 2% potassium aluminium sulphate, ferrous sulphate and zirconium oxychloride separately to study the combined effect of irradiation and mordanting on colour values and fastness properties.

**Results:** Pretreatment of cotton fabrics with gamma irradiation resulted in improvement in colour strength compared to untreated sample. Among the mordants potassium aluminium sulphate combined with gamma irradiation showed a considerable improvement in colour strength as compared to other mordants. Washing, rubbing and perspiration fastness was found to be in the acceptable range for all the dyed samples while the zirconium oxychloride mordanted samples showed good light fastness.

**Conclusion:** The colour strength, colour fastness ratings and shades obtained on cotton demonstrate the potential use of the extracts of *Nicotiana tabacum* as an eco-friendly natural dye on textile substrates.

**Key Words:** Colour fastness, gamma irradiation, mordants, natural dye, *Nicotiana tabacum*

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### **I. Introduction**

Natural dyes have been part of man's life, since time immemorial. The age-old art of dyeing with natural dyes was common in India. The earliest written record of the use of natural dyes was found in China dated 2600 BC<sup>1</sup>. Dyeing of textiles with synthetic dyestuff is characterized by its negative impact on the environment, dyers as well as end users. Therefore, interest in natural dyes has increased considerably on account of their high compatibility with environment, relatively low toxicity and allergic effects.

Natural dyes contain natural colouring matter, which are neither carcinogenic nor hazardous to environment. The colours are soothing to eyes and appealing, the dyes being natural are environmentally friendly and biodegradable. Some dyes even have some therapeutic values for which the raw materials find use in ayurvedic medicines. Dyeing with natural dyes has not achieved wide acceptability in organized sector due to their limited availability, limited shades and lack of standard procedures for dyeing, difficulty in reproducibility as well as lack of scientific knowledge on compatibility and chemistry of dyes<sup>2</sup>.

Most of the natural dyes have no substantivity for the fibre and are required to be used in conjunction with mordants. Mordants help in binding of the dyes to fabric by forming a chemical bridge from dye to fiber, thus improving the staining ability of a dye along with increasing its fastness properties. Metal ions of mordants act as electron acceptors for electron donors to form co-ordination bonds with the dye molecule, making them insoluble in water<sup>3</sup>. Surface modification of fibers for various applications is considered as one of the best methods for modern textile finishing processes especially for improving the dyeability of fabrics. Various treatments such as ultraviolet, ultrasonic and gamma radiation are being used to improve the colour strength and fastness properties of fabric dyed with natural dyes<sup>4-6</sup>.

Textiles substrates, when exposed to low doses of gamma radiation, the ionizing energy alters materials at the molecular level without changing their macro-structure<sup>7</sup>. This structural alteration increases the available surface area, accelerating moisture absorption and allowing the larger dye molecules to diffuse evenly into the

fiber core<sup>8</sup>. Thus, irradiated fabrics result in noticeably higher dye exhaustion yields and more vibrant shades compared to non-irradiated textiles even with reduced mordants / auxiliaries and enhanced colour fastness<sup>9</sup>.

*Nicotiana tabacum*, commonly known as tobacco is grown across the globe as a commercial crop in tropical and subtropical regions, primarily used in smoking and chewing products. The presence of carotenoids and polyphenols in the leaves and other parts of the plant has led researchers to consider it in dyeing studies. Various carotenoids, including lutein, neoxanthin, and violaxanthin, are known to be present along with polyphenols which could be used as dyes for dyeing cellulose fibers<sup>10,11</sup>. The use of tobacco waste as natural dye source and antibacterial agent for cotton fabrics is also reported<sup>12</sup>.

In the present study an attempt is made to extract the dye from the leaves of *Nicotiana tabacum*, dye cotton fabric and to study the influence of gamma irradiation and mordants on the colour strength and colour fastness properties.

## II. Material and Methods

### Material:

Commercially scoured and bleached cotton fabric (120 gsm, plain weave) was used. Tobacco leaves were procured from a local store in Bangalore. Laboratory grade sodium hydroxide, tannic acid, potassium aluminium sulphate, ferrous sulphate and zirconium oxychloride were used.

### Extraction of natural dye from tobacco leaves:

The dried tobacco leaves were crushed to powder form before being used for dye extraction. The dye extraction was performed using distilled water maintaining the ratio of 1:20. The extraction conditions were optimized by maintaining duration of 30, 45, 60, 75 and 90 min, temperature 60, 70, 80, 90 and 100°C, pH 3, 5, 7, 9 and 12. The extracts obtained were used for dyeing cotton and the K/S values were calculated and extraction conditions were optimized viz. boiling for 60 min at pH 9.

### Optimization of dyeing conditions:

The dyeing conditions were optimized by varying the duration, temperature and pH. The duration of dyeing maintained was 30, 45, 60, 90 and 120 min, temperature maintained was 60, 70, 80, 90 and 100°C and pH was 5, 7, 9, and 11 while the dye concentration maintained was 10% and liquor ratio was 1:30 for all the dyeings.

### Pretreatment of cotton with gamma irradiation:

Samples of scoured and bleached cotton fabrics (50cm x 50cm) were exposed to gamma radiation to the absorbed doses of 1,2,3,4,5 kGy respectively using Co-60 gamma irradiator at Microtrol Sterilization Services Pvt. Ltd, Bangalore.

### Mordanting and Dyeing:

Un-irradiated and gamma irradiated cotton fabrics were treated with different mordants such as potassium aluminium sulphate, ferrous sulphate, and zirconium oxychloride separately. For mordanting, cotton fabric was impregnated in a 2% (owf) mordant bath at a liquor ratio of 1:30, at 70°C for 30 min before dyeing. Un-irradiated and gamma irradiated cotton fabrics with and without mordanting were dyed in open bath beaker dyeing machine maintaining a liquor ratio of 1:30 and pH 9, at 90°C for 60 min with dye concentration of 10%. After dyeing, the dyed samples were rinsed with cold water, soaped with 2 gpl of soap solution, at 60°C for 10 min, and finally rinsed and dried.

### Determination of colour strength and colour coordinates:

Colour was evaluated by means of K/S and CIELAB colour coordinates with illuminant D65/10° observer on Gretag Macbeth Colour Eye 7000 A Spectrophotometer by measuring the reflectance of the dyed samples. The K/S values were determined using the Kubelka Munk equation.

$$K/S = (1 - R)^2/2R$$

where,  $R$  is the observed reflectance at  $\lambda_{\text{max}}$ ,  $K$  is the absorption coefficient, and  $S$  is the light scattering coefficient.

### Determination of colour fastness properties:

The colour fastness to washing, light, crocking and perspiration of the dyed samples were determined according to ISO 105-C10:2006<sup>13</sup>, ISO 105-B02:2014 (xenon-arc apparatus)<sup>14</sup>, ISO 105-X12:2016<sup>15</sup> and ISO 105-E04:2013<sup>16</sup> methods respectively.

### III. Results and Discussion

#### Effect of extraction conditions

It was observed that with the increase in extraction time, there was increase in the yield of dye expressed as colour strength of the dyed fabric. But the rate of increase gradually decreased after 60 min of extraction (Figure 1). The ideal temperature for extraction of the dye was found be boiling as the maximum K/S value was recorded at boiling temperature (Figure 2). Maximum colour strength was recorded for extraction done at pH 9 (Figure 3). Therefore, the optimum conditions for extraction of dye from tobacco leaves were boiling for 60 min at pH 9.

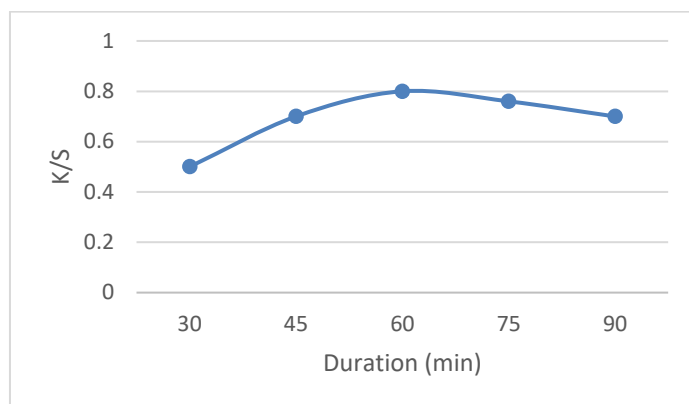


**Figure 1.** Effect of duration on dye extraction

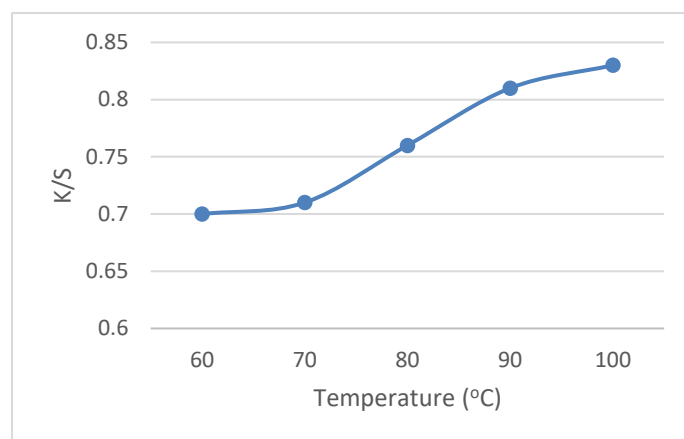


**Figure 2.** Effect of temperature on dye extraction

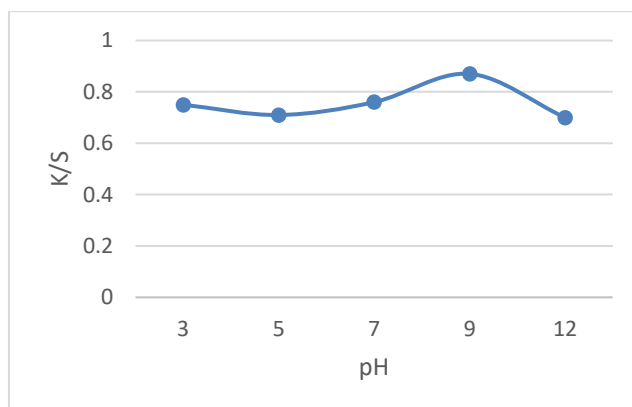


**Figure 3.** Effect of pH on dye extraction

#### Effect of Dyeing Conditions

The influence of dyeing conditions viz. duration, temperature and pH are given in figures 4-6. Increase in the duration of dyeing resulted in an increase in the K/S values upto 60 min after which it did not show any further improvement. The increase in the dye bath temperature also showed an increase in the colour strength upto 90°C

after which it did not show any significant increase. The decrease in colour strength after 60-90 min of dyeing may be attributed to desorption of the dye molecules as a consequence of over dyeing. The maximum colour strength was observed for the samples dyed at pH 7.

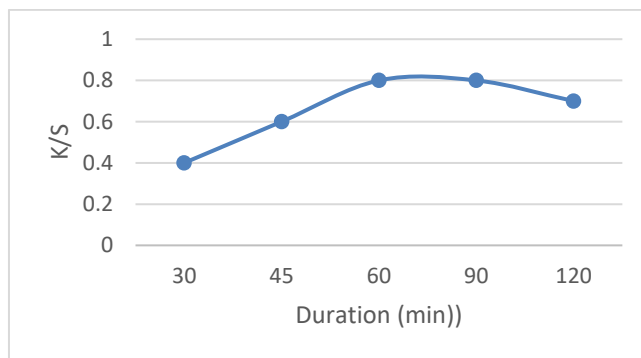


**Figure 4.** Effect of dyeing duration on colour strength of cotton fabrics

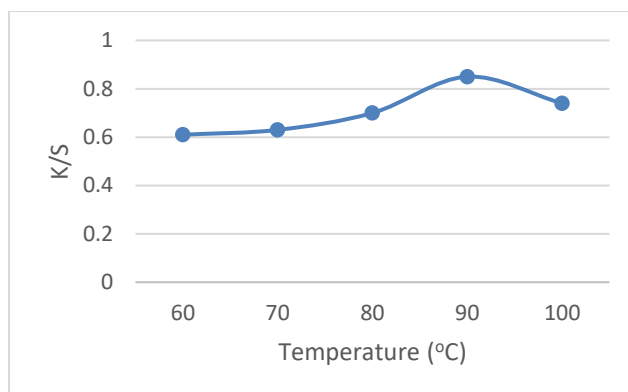


**Figure 5.** Effect of dyeing temperature on the colour strength of cotton fabrics

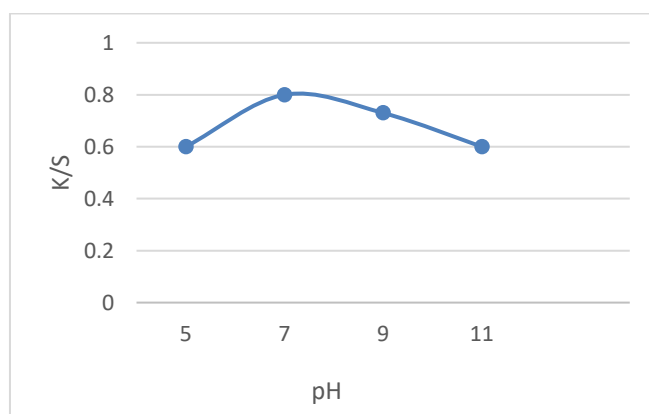


**Figure 6.** Effect of dye bath pH on colour strength of dyed cotton fabrics

#### Effect of irradiation and mordanting on colour strength and colour coordinates

The K/S values and CIE L a b coordinates of unirradiated and irradiated cotton fabrics without mordant and with mordant treatment are shown in Table 1. It is observed that the pretreatment of irradiation of cotton fabrics has resulted in the increase in K/S values and a change in the a\* and b\* coordinates. Previous studies also reported an increase in colour strength when gamma irradiated cotton fabrics were dyed with natural dyes. The structural changes induced by irradiation enhances the dye uptake and fixation of dye in the fibres<sup>7,17</sup>. Scanning electron microscopy images of unirradiated and irradiated cotton fabric samples are shown in figure 7. The unirradiated cotton fabric sample is characterized by a comparatively smooth undisturbed surface while the

irradiated cotton fabrics showed slight morphological changes on the surface of the fibres. Gamma radiation is known to significantly affect the structural and morphological properties of cotton fibers<sup>8</sup>. It is also reported that gamma irradiation, especially higher doses results in the formation of surface cracks along the cellulose fibers, reflecting significant structural degradation<sup>18</sup>. As the absorbed dose is limited to only 5kGy significant surface cracks are not evident from the images. It is observed that the colour strength of the gamma irradiated cotton fabrics increased considerably with the use of mordants as compared to the un-irradiated dyed sample. All the mordants used in the study also have resulted in improved K/S values and have influenced the shades obtained which is visible in the images provided in Table 1. Combined effect of irradiation and mordanting is visible with the enhanced colour strength as compared to un-irradiated and unmordanted samples. An increase in the a and b values was observed in case of all the mordants resulting in redder and yellower samples while ferrous sulphate resulted in lowered a and b values leading to duller samples due to the formation of iron-tannate complexes before dyeing as compared to unmordanted samples. Among the mordanted samples aluminium potassium sulphate showed maximum colour strength followed by zirconium oxychloride and ferrous sulphate. The aluminium ions form strong coordination bonds with the enhanced carboxylic groups and hydroxyl groups due to irradiation and further bind to the functional groups of the dye molecules resulting in higher colour strength compared to unirradiated samples. The decrease in the L value (lightness) on irradiation and mordant treatment indicates darker samples. All the dyed samples lie in the yellow red quadrant of the colour space diagram as observed by the a\* and b\* values which are positive. Overall lower doses of irradiation resulted in increased K/S values as compared to higher absorbed doses. 1 kGy in case of ferrous sulphate and zirconium oxychloride and 2kGy in case of potassium aluminium sulphate mordanted samples resulted in enhanced colour strength which is in agreement with the earlier studies<sup>19</sup>.

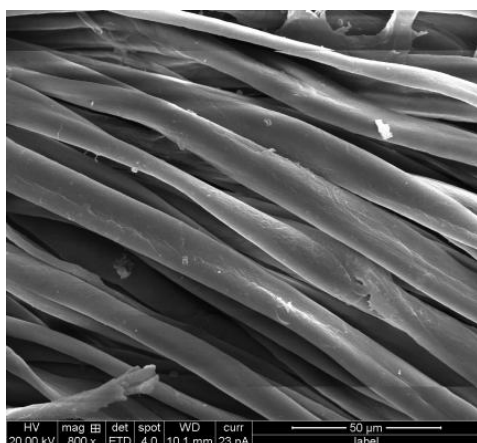
#### Effect of gamma irradiation and mordanting on fastness properties

The fastness properties of the dyed cotton fabrics pretreated with gamma irradiation and different mordants along with the untreated dyed cotton fabrics is presented in table 2. *Nicotiana tabacum* leaf extract showed fair to good fastness properties on cotton except light fastness (2-3) even without irradiation and mordanting. Slight improvement in colour fastness properties was observed in fabrics pretreated with irradiation which showed further improvement with mordanting. Washing fastness and perspiration fastness ranged from 3-4 to 4-5 and rubbing fastness ranged from 4 to 4-5 whereas light fastness ranged from 2-3 to 4. Among the mordants used, considerable improvement in colour fastness properties especially light fastness was observed with zirconium oxychloride. It is reported that zirconium acts as a chemical bridge, coordinating strongly with the hydroxyl groups of cotton fibres and the electron-donating atoms in natural extracts. It acts as a protective shield against photolytic degradation without causing environmental damage unlike other metal mordants, preventing UV light from cleaving the chromophores of natural plant extracts<sup>20</sup>.

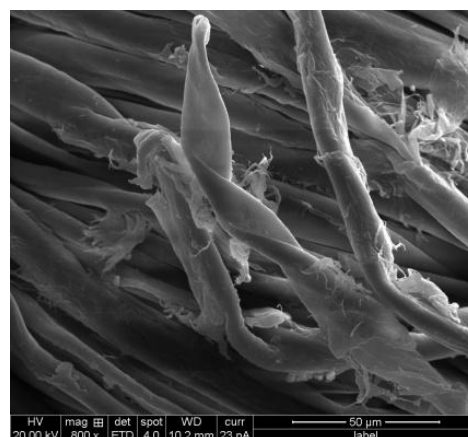
**Table 1.** Colour strength and colour coordinates of pre-treated dyed cotton fabrics

| Mordant                      | Irradiation dosage | K/S  | L*    | a*   | b*    | C     | h     | Dyed sample                                                                           |
|------------------------------|--------------------|------|-------|------|-------|-------|-------|---------------------------------------------------------------------------------------|
| Nil                          | Nil                | 2.43 | 67.84 | 5.36 | 21.39 | 22.05 | 75.93 |  |
|                              | 1 kGy              | 3.18 | 67.01 | 5.31 | 22.26 | 22.88 | 76.59 |  |
|                              | 2 kGy              | 2.62 | 68.57 | 4.7  | 20.4  | 20.95 | 77    |  |
|                              | 3 kGy              | 3.09 | 66.43 | 5.49 | 21.63 | 22.31 | 75.75 |  |
|                              | 4 kGy              | 3.59 | 66.39 | 5.51 | 23.43 | 24.07 | 76.76 |  |
|                              | 5 kGy              | 2.97 | 67.35 | 5.15 | 21.4  | 22.01 | 76.45 |  |
| Potassium aluminium sulphate | Nil                | 4.02 | 67.57 | 5.3  | 23.8  | 24.4  | 77.48 |  |
|                              | 1 kGy              | 6.07 | 64.48 | 6.49 | 27.7  | 28.46 | 76.8  |  |
|                              | 2 kGy              | 7.64 | 62.92 | 7.02 | 30.0  | 30.8  | 76.82 |  |
|                              | 3 kGy              | 6.07 | 64.63 | 6.37 | 27.53 | 28.26 | 76.96 |  |
|                              | 4 kGy              | 5.36 | 66.12 | 5.99 | 27.03 | 27.69 | 77.49 |  |
|                              | 5 kGy              | 6.89 | 62.88 | 6.84 | 28.87 | 29.67 | 76.66 |  |
|                              | Nil                | 2.83 | 57.00 | 4.58 | 8.88  | 9.99  | 62.72 |  |

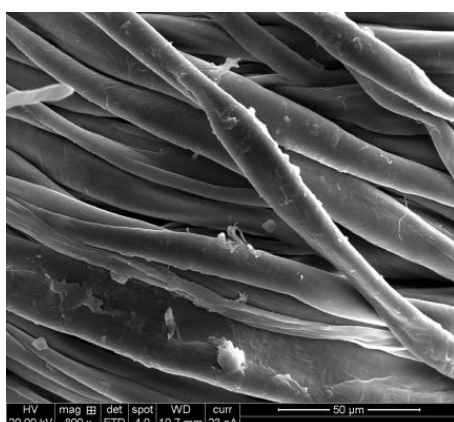
|                       |       |      |       |      |       |       |       |                                                                                     |
|-----------------------|-------|------|-------|------|-------|-------|-------|-------------------------------------------------------------------------------------|
| Ferrous sulphate      | 1 kGy | 3.55 | 53.58 | 4.43 | 8.9   | 9.94  | 63.52 |  |
|                       | 2 kGy | 3.26 | 54.62 | 4.17 | 8.4   | 9.38  | 63.57 |                                                                                     |
|                       | 3 kGy | 2.97 | 55.98 | 4.21 | 8.93  | 9.87  | 64.73 |                                                                                     |
|                       | 4 kGy | 3.27 | 54.66 | 4.0  | 8.78  | 9.69  | 64.97 |                                                                                     |
|                       | 5 kGy | 3.01 | 55.79 | 4.0  | 8.57  | 9.49  | 64.59 |                                                                                     |
| Zirconium oxychloride | Nil   | 4.04 | 66    | 5.9  | 26.42 | 27    | 77.4  |  |
|                       | 1 kGy | 4.36 | 65.1  | 6.11 | 26.73 | 27.4  | 77.12 |                                                                                     |
|                       | 2 kGy | 4.22 | 66.13 | 5.98 | 26.6  | 27.27 | 77.32 |                                                                                     |
|                       | 3 kGy | 4.20 | 66.25 | 5.79 | 26.06 | 26.7  | 77.47 |                                                                                     |
|                       | 4 kGy | 4.15 | 66.18 | 5.87 | 26.41 | 27    | 77.46 |                                                                                     |
|                       | 5 kGy | 4.21 | 66.11 | 5.94 | 26.68 | 27.34 | 77.45 |                                                                                     |



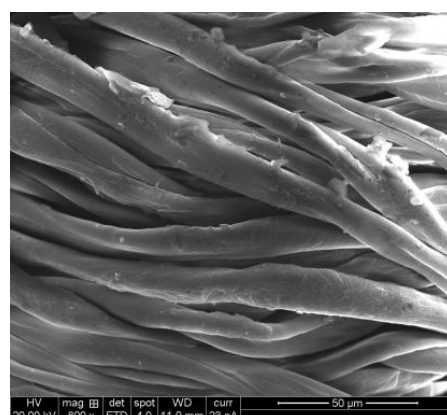
Unirradiated



1kGy



2 kGy



3kGy

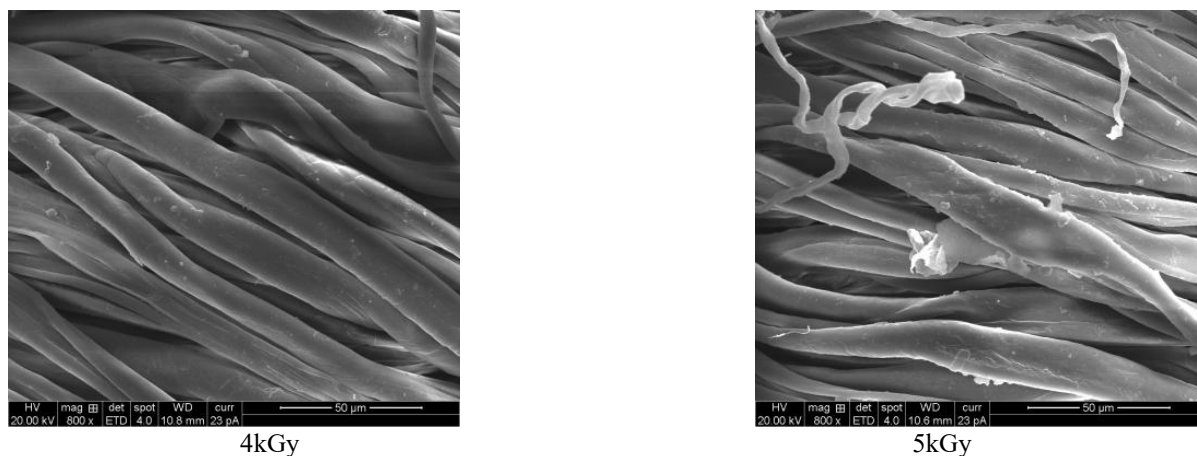


**Figure 7.** SEM images of the surface morphology of fabric samples before and after irradiation.

**Table 2.** Fastness properties of cotton fabrics dyed with *Nicotiana tabacum* leaf extract

| Mordant                      | Absorbed Dose | Light fastness | Wash fastness |     | Perspiration fastness |    | Croaking fastness |     |
|------------------------------|---------------|----------------|---------------|-----|-----------------------|----|-------------------|-----|
|                              |               |                | CC            | CS  | CC                    | CS | Dry               | Wet |
| Nil                          | Nil           | 2-3            | 3-4           | 5   | 3-4                   | 5  | 4                 | 4   |
|                              | 1kGy          | 3              | 4             | 5   | 4                     | 5  | 4-5               | 4   |
|                              | 2kGy          | 2-3            | 4             | 5   | 4                     | 5  | 4-5               | 4   |
|                              | 3kGy          | 3              | 4             | 5   | 4                     | 5  | 4-5               | 4   |
|                              | 4kGy          | 3              | 3-4           | 5   | 4                     | 5  | 4-5               | 4   |
|                              | 5kGy          | 3              | 3-4           | 5   | 4                     | 5  | 4-5               | 4   |
| Potassium aluminium sulphate | Nil           | 2              | 3-4           | 4-5 | 3-4                   | 5  | 4                 | 4   |
|                              | 1kGy          | 3              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 2kGy          | 3              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 3kGy          | 2-3            | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 4kGy          | 2-3            | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 5kGy          | 2-3            | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
| Ferrous Sulphate             | Nil           | 3              | 4             | 5   | 4                     | 5  | 4-5               | 4   |
|                              | 1kGy          | 3-4            | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 2kGy          | 3              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 3kGy          | 3-4            | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 4kGy          | 3              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 5kGy          | 3              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
| Zirconium Oxychloride        | Nil           | 4              | 4             | 5   | 4                     | 5  | 4-5               | 4   |
|                              | 1kGy          | 4              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 2kGy          | 4              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 3kGy          | 4              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 4kGy          | 4              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |
|                              | 5kGy          | 4              | 4-5           | 5   | 4-5                   | 5  | 4-5               | 4   |

#### IV. Conclusion

It was found from the study that *Nicotiana tabacum* leaf extract can be successfully used for dyeing cotton without mordants. Gamma irradiation pretreatment used in the present study has resulted in enhanced colour strength and colour fastness properties. Mordants also resulted in improved colour strength and colour fastness, exhibiting excellent fastness to washing, rubbing, perspiration and good fastness to light besides leading to various shades. The combined effect of irradiation and mordanting was visible with the enhanced colour strength and fastness properties. As the dye extraction and process of dyeing involves simple procedures and does

not involve the use of harmful chemicals the entire process is environment friendly. Hence *Nicotiana tabacum* can be considered a potential renewable source of natural dye for dyeing cotton.

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