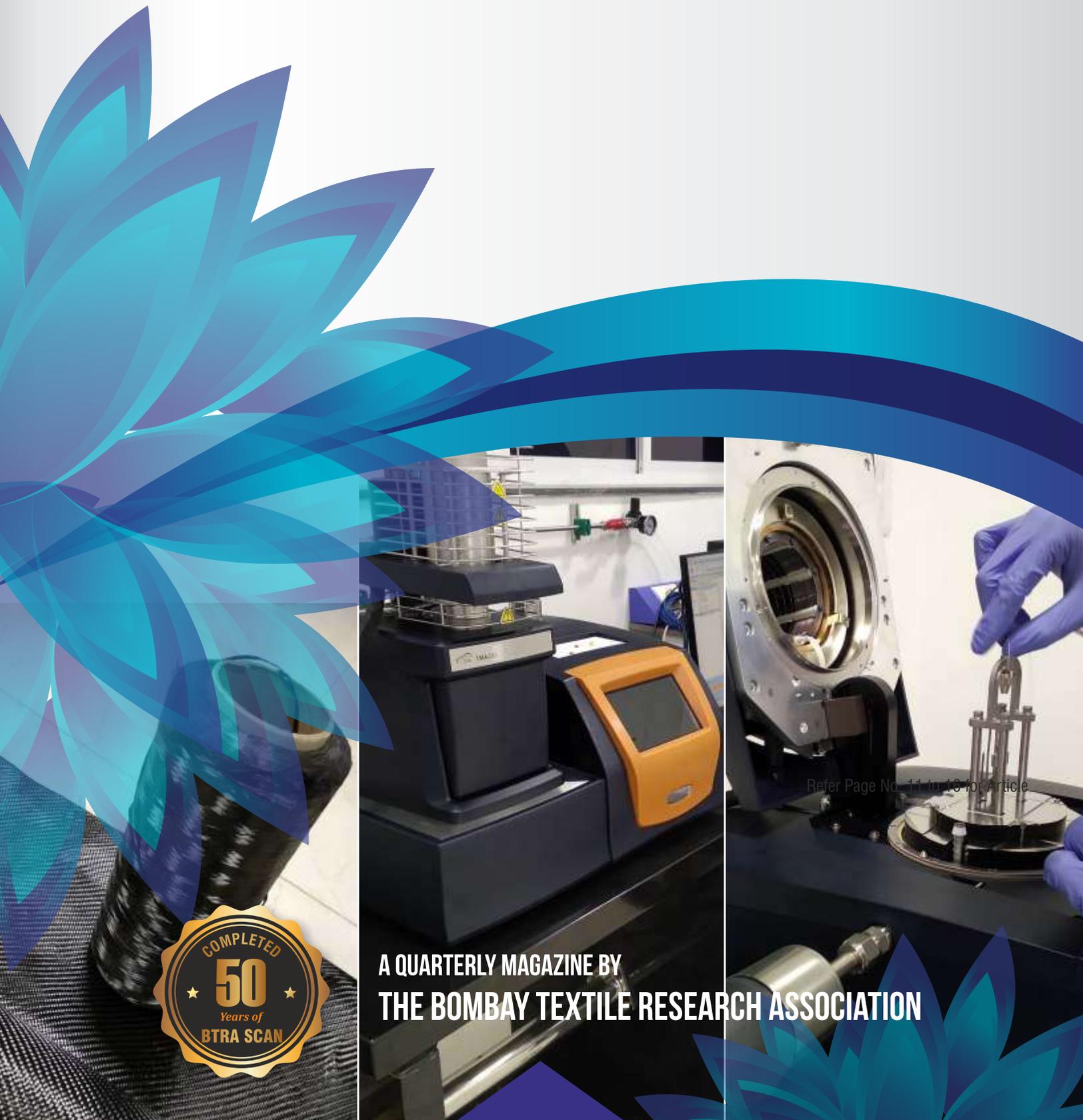


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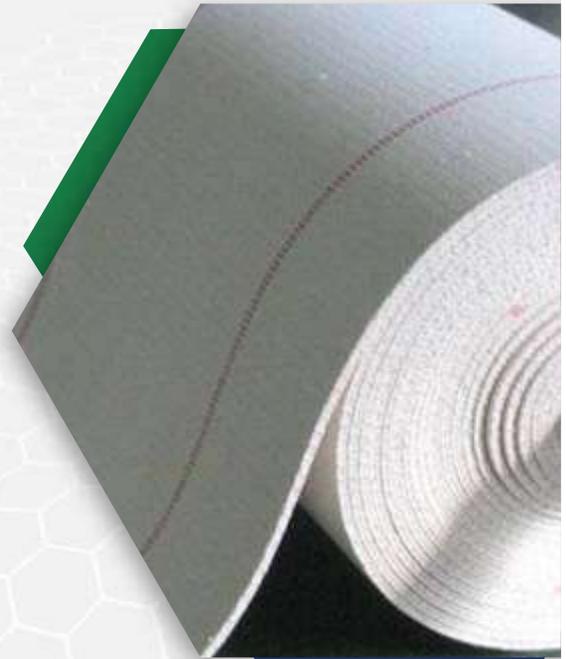
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EDITOR'S DESK

Dear Readers,

Greetings!!

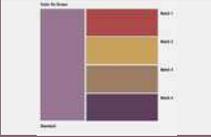
Research with persistent and focused efforts lead to a positive result. Fostering research and providing a platform to publish quality research papers and related articles has been a continuous effort of BTRA Scan. It is time for us to continue work hard to help the journal in climbing up the ranking ladder. In continuation to this effort, I am delighted to present to our readers the 3rd issue of 51st Edition of BTRA SCAN.

This issue has papers from the different domains such as natural dyeing of silk, textile effluent treatment by emulsion method, carbon fiber cost component and fume toxicity. Now we are open for authors from outside so researchers can send their original articles, case studies, research reviews or empirical contributions for publication in our journal.

I thank my associate editor Dr. Prasanta K. Panda and the entire publishing team for all their support. Together we would work towards making the journal a truly influential publication. Comments and suggestions are always welcome.

Our sincere thanks to all the reader and contributors for their support and interest.

T V Sreekumar, PhD
Director, BTRA

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Color Removal from Textile Effluent using Emulsion System

Babita U. Chaudhary, Sandip Todkar & Ravindra D. Kale*

Department of Fibers and Textile Processing Technology, Institute of Chemical Technology, Mumbai

Abstract

India's domestic clothing and textile sector produce 5% of the country's GDP, 7% of industry output in value terms, and 12% of the country's export revenues. The primary threat faced by the textile industry is the color of the water that is left after dyeing. In the present study, water in oil nanoemulsion was used to remove the color from textile effluent. Nano-emulsion based on cyclohexanol was prepared using a high-speed homogenizer followed by ultrasonication. The emulsion, in general, was prepared using dye effluent water as the aqueous phase and cyclohexanol as the oil phase, and Beisol DEP as an emulsifier. The prepared emulsion was used to treat Reactive Blue 21 and Reactive Black 5. The treatment ratio of 1:1, 1:3, 1:5, 1:7, 1:10 were tried. The treated effluent was tested for color removal efficiency, Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD). Also, this study investigated the reusability of emulsion and solvent recovery. Emulsion characterization such as particle size and zeta potential was also carried out. It was found that there was about 95% decolourisation dye in effluent and the reusability of the solvent decreased by 7-10% after each cycle.

Keywords:

Dye removal; Effluent treatment; Emulsion system; Reactive Dye; Reusability

Citation

Babita U. Chaudhary, Sandip Todkar & Ravindra D. Kale - "Color Removal from Textile Effluent Using Emulsion System", *BTRA Scan* - Vol. LI No. 3 July 2022, Page no. 1 to 3

1.0 Introduction

The textile industry stood as the largest in all amongst industries in the Indian economy for several decades. Even today, the textile industry is a significant contributor to India's export business, around 13 percent of all exports. The textile business employs more than 65 million people directly and indirectly. Total India's export in textiles from 2016-2017 was around 40 billion USD [1]. The textile business in India exports the most considerable textile material and is also responsible for producing a large amount of effluent, which is quite challenging to treat and not cost-effective.

The effluent generated by the textile industry, which consumes a very high amount of dyes and pigments, causes high coloration to the effluent. The dyes and pigments have complex chemical structures, large molecule sizes, and toxic nature. These pigments and dyes are responsible for the dangerous effect on human and aquatic life [2]. The high concentrations of dyes cause mutagenic effects on the marine ecosystem. The colors and pigments mostly have complex aromatic structures. These aromatic structures are difficult to degrade and require significant chemical oxygen demand (COD) [3].

During dyeing, some amount of dye remains in the dye bath, and also unfixed dyes come out from the fabric during the

washing operation. This unfixed dye is high in concentration. When mixed with the effluent causes high coloration, which has highly complex structures and is challenging to remove through conventional biological water treatment processes is also resilient to microorganisms when present in a mixture with various auxiliaries [4]. This effluent consists of highly concentrated dyes and auxiliaries used in various processing stages of textile material. The effluent contains few metal traces such as Cu, Zn, Cr, and As are capable of causing several health issues like nausea, irritation to the skin, dermatitis, hemorrhage, and ulceration of human skin [5].

Several treatments are employed to treat effluent, which follow primary, secondary, and tertiary treatments. These treatments include flocculation, coagulation, sedimentation, aerobic activated sludge, aerated lagoons, reverse osmosis (RO), trickling filter, electrodialysis, and nanofiltration [6][7]. But no single method can be used to remove more than one class of dye or complete removal of a particular dye from effluent. Also, most methods are too costly and hence are not feasible to be used in the current scenario, or the sludge generated is high [8].

In this work, we have developed a simple emulsion using cyclohexanol as the oil phase and water as the aqueous phase and made water in the oil emulsion using an emulsifier, which can remove the color from effluent and then it can be taken up for further treatment. The emulsion technique is an

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easy way the removal of dyes from wastewaters. It transports them into desired phases, where the pollutants can be concentrated 10 to 100 times. There are no sludge generation less operational and energy costs than conventional effluent treatment. The emulsion separation technique has been regarded as membrane technology with considerable potential for various applications.

2.0 Materials and Method

2.1 Materials

Cyclohexanol was procured from Amrut Chemicals, Mumbai, and CHT Chemicals supplied surfactant (Beisol DPH). Colourtex industries, Mumbai, provided various dyes. All chemicals were used without any further purification.

2.2 Methods

2.2.1 Preparation of Emulsion

Sets of the experiment were performed to obtain a stable emulsion using cyclohexanol and effluent water and an emulsifier. The cyclohexanol to water ratio was tried from 90:10, 80:20, 70:30, 60:40, and 50:50, and surfactant concentration was varied from 5, 10, and 20%. The ratio of O/W 90:10 and surfactant concentration of 20% gives excellent stability compared to other O/W ratios and surfactant concentrations. This stable emulsion was utilized to treat reactive dye effluent.

A known amount of Beisol DEP was added to cyclohexanol using a magnetic stirrer at 500 rpm at room temperature. The aqueous phase water was added dropwise by using a high-speed homogenizer at a speed of 12000 rpm for 15 to 20 min, followed by ultrasonication for 15 min. The oil to water ratio was kept at 90:10.

2.2.2 Use of Emulsion in Effluent Treatment

The prepared emulsions were added to Reactive Blue 21 (RB 21) and Reactive Black 5 (RB 5) effluent in the ratio of 1:1, 1:3, 1:5, 1:7, 1:10 and three different concentrations of effluent dye solution, i.e., 125, 250, and 500 mg/L were used for treatment. The selection of both dyes is that they are most widely used and are difficult to degrade by bacterial or oxidative methods [9].

The physical properties of emulsion like particle size and zeta potential were characterized by using SHIMADZU SALD 7500 Nano, Japan, and Zetasizer Ver. 7.11, Malvern, UK. To check the dye concentration after treatment the absorbance of treated effluent was measured by using a UV-vis spectrophotometer (SHIMADZU 1800 UV vis, Japan). The optimized treated dye effluent of RB 21 and RB 5 of concentration 250 mg/L and treatment ratio 1:10 was tested for COD and BOD using Hatch DRB 200 and Hatch BOD Track II., USA, respectively. The cyclohexanol emulsion was reused to treat the effluent of Reactive Black 5 dye having a concentration of 250 mg multiple times.

3.0 Result and Discussion

3.1 Characterization of emulsion

The colorless emulsion obtained showed a particle size of about 30nm. It is said that the lower the particle size higher is the stability of the emulsion [10]. The Zeta potential values between -25 mV and -35 mV are sufficient to avoid the separation of nanoemulsion into different phases, leading to good stability [10]. The zeta potential was found to be -32.2 mv for emulsion after preparation.

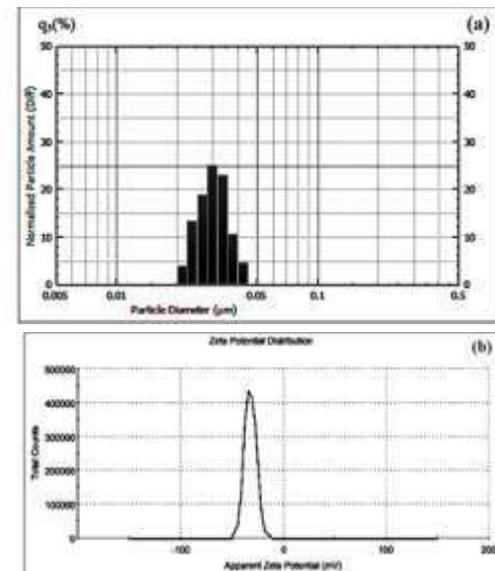


Figure 1 -The particle size (a) and zeta potential (b) graph of the emulsion

3.2 The absorbance of treated effluent by using UV-vis spectrophotometer

Fig 2. shows the results of % decolorization for RB 21 and RB 5 at different concentrations treated with cyclohexanol emulsion. The effluent was decolorized by more than 95% for a ratio of 1:10 in a span of 6 h.

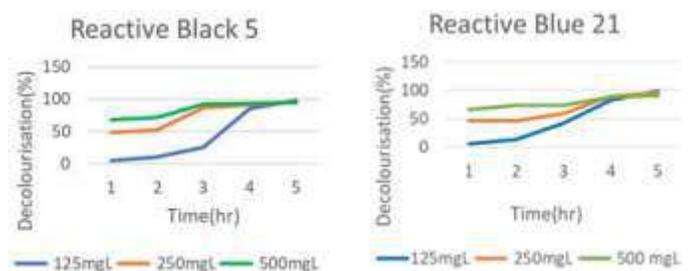


Figure 2 - % Decolourisation of RB 5 and RB 21 after about 5 h of treatment

3.3 COD and BOD analysis

It showed 167 mg/L and 139 mg/L values compared to BOD values of 4 mg/L and 8 mg/L of standard untreated dye effluent of RB 21 and RB 5. It showed 229 mg/L and 258 mg/L values compared to COD values of 150 and 158 mg/L of RB 21 and RB 5. There was a considerable increase in BOD and COD values in both dyes. The increased values were due to cyclohexanol and a high amount of surfactant.

3.4 Visual analysis of the treated samples

As can be seen from fig. 3a and 3b all the ratios of RB 21 and RB 5 give almost complete decolorization in 6 h. Different ratios of emulsion and effluent were tried. It can be seen that even with minimum usage of emulsion, i.e., 1ml for 10 ml of effluent, a complete decolorization is there for both the dyes.

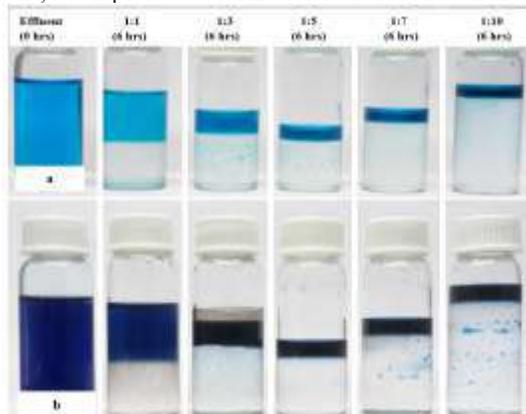


Figure 3 - visual confirmation of dyes a. RB 21 and b. RB 5 removal from the effluent in all concentrations

3.5 Recovery and reusability of cyclohexanol

The cyclohexanol emulsion was reused to treat the effluent of Reactive Black 5 dye with 250 mg multiple times, as can

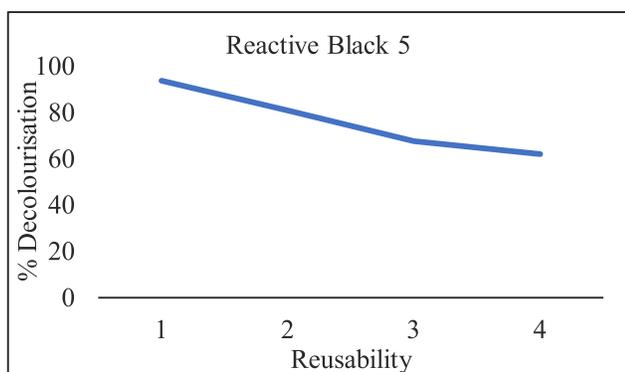


Figure 4 - Reusability of cyclohexanol emulsion

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be seen in fig 4. The color removal percentage reduced to 62% after being used 4 times. The efficiency decreases may be due to the saturation of water present in the emulsion with the dye particles. After each use, the reactivity of the emulsion decreased by 8–10%.

3.6 A trail on Industrial Effluent

An unknown effluent supplied by Atul Industries (Valsad, India) was treated with cyclohexanol emulsion in the ratio of 1:5 for 12 h and 48 h. The results are shown in figure 5, and it is clear that after 12 h, slight traces of dyes were present in the water in both effluent while the 48 h treatment showed the complete removal of color from the effluent.

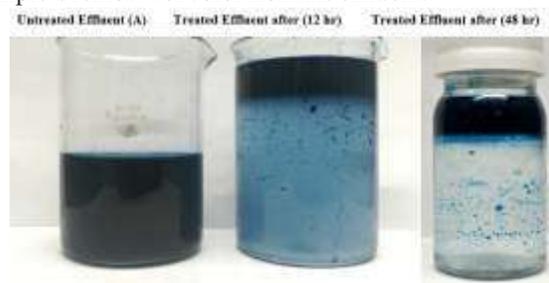


Figure 5 - Treatment of industrial effluent

4. Conclusion

Water in oil emulsion was successfully carried out using cyclohexanol as an oil phase, effluent as an aqueous phase, and Beisol DEP as a surfactant. The zeta potential was -32.2, indicating that the emulsion was stable. Also from UV-vis spectroscopy, we can see that more than 95% decolorization is there. There was an increase in COD and BOD values due to the cyclohexanol used, but it can be reduced in the subsequent stages of effluent treatment. Also, if the emulsion is recovered, the value will go down. The decolorization was more than 95% after 6 h of treatment. It can be implemented in the industrial process after the primary treatment of effluent.

5. Acknowledgment

The authors would like to express sincere gratitude to the World Bank Funded TEQIP-III CoE Process Intensification to provide financial support

Better Dyeability in Natural Dyeing of Silk using Rare Earth (RE) Salts as Mordant

Archana Gangwar and Padma S Vankar*

The Bombay Textile Research Association, L B S Marg, Ghatkopar (W), Mumbai 400086

Abstract

Natural dyeing requires two main features- a natural colorant and a mordant which can fix the natural colorant to the fabric. Selection of an appropriate metallic compounds as mordants is essential as well as crucial as there is a natural dye and mordant compatibility factor. Mordant is required for dyeing natural fibers with natural dyes. This paper presents the application of rare earth (RE) salts as mordant for the dyeing of pure silk fabrics by two different modes of mordanting using a natural dye—*Rubia cordifolia*. The influence of pre-and post -mordanting on silk dyeing were explored. The rare earth mordant dyeing was found to improve dye-uptake, the fixation of dyestuff, and thus causing better adherence of the colorant and good colour fastness. Another speciality of RE salts is that they provide different hues to the fabric with *Rubia* dye extract.

Through this study we wish to report that with the use of rare earth salts, there has been manifold advantages such as better dye uptake, better fastness properties, low temperature dyeing, low energy consumption, and above all would have lesser pollution load in the effluent, thus proving them to be ecofriendly and safe mordant.

Keywords

Rare earth salts; Lanthanum carbonate; Yttrium oxide; Cerium nitrate; Premordanting; Postmordanting; *Rubia cordifolia*.

Citation

Archana Gangwar and Padma S Vankar - Better dyeability in Natural dyeing of Silk using Rare earth (RE) salts as mordant, *BTRA Scan* - Vol. LI No. 2 July 2022, Page no. 4 to 9

1.0 Introduction:

With natural dyes mordanting with metal salts is an integral part of the dyeing process. These are metallic salts which are applied onto the fabric before, or after the dyeing step (they are called pre and post-mordanting respectively). Conventionally salts such as alum, copper sulphate, ferrous sulphate, and tin chlorides metal salts are being used with natural dyes. Other methods of mordanting could involve the use of biomordants which are derived from plants such compounds are polyphenolic commonly called tannins and tannic acid as well as enzymes[1-3]. The use of some metal accumulating plants such as *Eurya acuminata* DC var *euprista* Karth in natural dyeing has been reported by Vankar et al [4]. This plant inherently contained Aluminium which was responsible for dye adherence.

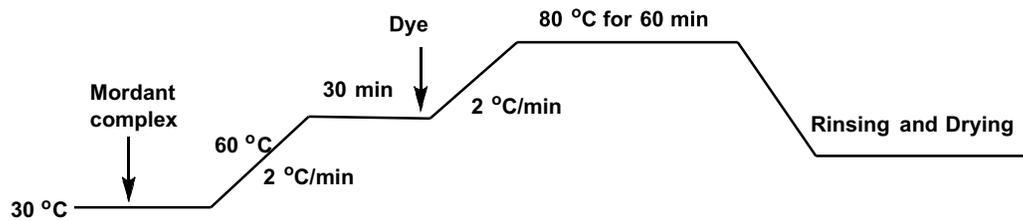
The role of a mordant is well defined. It acts like a bridging head between the colorant and the fabric and thus is responsible for better dye adherence. Mordants help to bind

the dyestuff to the fibre. There are many mordants commonly used in natural dyeing and each one will give a different shade from a particular dyestuff as per its chelating capacity. Depending on the oxidation state of the metal of the mordant, the dye chelation takes place[5].

Rare earth element (Rare Earth) salts are also called (RE salts); salts from lanthanide series such as lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd), promethium (Pm), samarium (Sm), europium (Eu), terbium (Tb), ytterbium (Yb), dysprosium (Dy), holmium (Ho), gadolinium (Gd), erbium (Er), thulium (Tm), lutetium (Lu), and with closely-related two elements such as yttrium (Y), scandium (Sc). The Rare earth (RE) are a unique group of elements. Their f electronic configurations, the hard Lewis acid character of their ions, and their large ionic radii render their coordination chemistry distinctly different from that of transition metal elements. Specifically, because of the large ionic size, a high coordination number is required unless sterically bulky ligands are utilized. The insignificant involvement of the f orbitals in chemical bonding means lanthanide–ligand interactions are primarily ionic, leading to

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Pre-mordanting



Post-mordanting

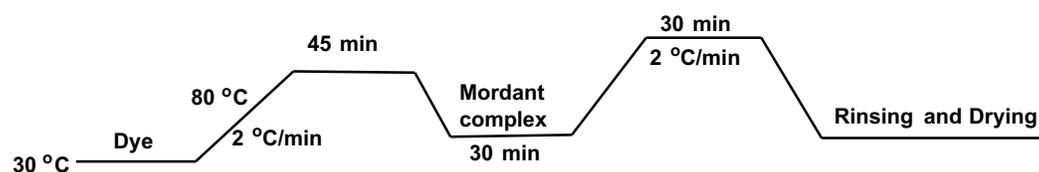


Figure 3 - Schematic representation of Pre and post mordanted dyeing

Rubia dyeing with RE salts (Pre and post mordanting)

The dyeing bath was heated to 60°C where it was maintained for a particular time (60 minutes). The silk fabrics were then rinsed with clean water at ambient temperature followed by being squeezed and dried. In the pre-mordanting method, the wet fabrics were first immersed in a solution of the mordant and heated to 60°C where they were kept for 30 minutes. After being added with the natural dyes the bath was heated to 80°C where the fabrics were treated for a particular time (45 minutes). Then the dyed samples were rinsed with clean water and dried via similar processes as mentioned above. In the post-mordanting method, the silk fabrics were first dyed in an aqueous solution containing a natural dye at 80°C for 30 minutes followed by being cooled to 60°C where the mordant was added. Then the fabrics were kept at 60°C for another 30 minutes. Subsequently, the dyeing bath was heated to 80°C again where the fabrics were dyed for a time in the range of 45-50 minutes. At last, the dyed fabrics were rinsed and dried. The effects of dyeing temperature and time on the dyeing of silk fabrics with the Rubia natural dye was studied as shown in figure-3. Silk swatched (RFD) were premordanted by 4% mordants such as alum (Conventional) and the rare earth salts 0.4%(Lanthanum carbonate, Yttrium oxide, and Cerium nitrate separately with citric acid in the ratio of (1:3). Mordanting was done at room temperature for 30 mins. After drying the samples were dipped in (Rubia 5% owf) dye bath and slowly the temperature was raised to 70-80°C over a period of 30 minutes, and stirring of the fabric was done for 45 minutes. After dyeing the samples were washed with water and dried. Similarly post mordanting was done after dyeing with similar concentrations of the mordant.

K/S Measurements

The dye uptake in the dyed silk swatches was obtained through the measurement of the light absorbances at the wavelength of maximum absorption, of the dye bath before and after dyeing with an ultraviolet-visible (UV-Vis) spectrophotometer. The dye uptake was calculated with the following equation:

$$\text{Dye uptake} = \frac{A_b - A_a}{A_b} \times 100\%$$

where A_b and A_a refer to the absorbance of the dye solutions at the beginning and the end of dyeing, respectively. The CIELAB colorimetric values including ΔE , L^* , a^* , b^* , C^* , and the color strength K/S of the dyed fabrics were measured by Premier Computer Color Matching System using illuminant D65 and 10 standard observer. K/S was calculated from the reflectance values K/S using the Kubelka-Munk equation as follows:

$$K/S = \frac{(1 - R)^2}{2R}$$

Where R represents the reflectance of the dyed fabric and K/S stands for the ratio of the absorption coefficient (K) to scattering coefficient (S). The higher the K/S value the greater the color strength.

The color fastness of washing of the silk fabrics was determined according to the standard ISO method ISO 105-C10 (2006). The measurement was carried out with both sample and standard silk fabrics that were sewn together and tested under the same conditions. The dyed silk fabric was washed at 40°C for 30 minutes in a non-ionic soap solution with material to liquid ratio of 1:50. All dyed fabrics were

then separately rinsed and dried. The colorfastness to washing levels, observed against a grey scale, were classified as numbers ranging from 1 and 5, which corresponds to poor to excellent fastness, respectively. Colorfastness to light was tested according to ISO 105-BO2 method.

3.0 Results and Discussion:

The dye uptake in the case of premordanted dyed silk swatch was from 52-58 % in the case of Cerium nitrate, 62-64% in the case of premordanted lanthanum carbonate, and 85-87% in the case of Yttrium oxide. This is in accordance with the K/S values.

The graph of K/S values shown in figure-4 is showing a bathochromic shift for yttrium oxide mordanted fabric with respect to alum mordanted. The plausible explanation could be that coordination through Y+3RE(III) could be 6,7,8,9 due to its ionic radius being close to 1.0, while for Al+3 coordination can be only 4,5,6 as the ionic radius is 0.53. Owing to the high positive charge, large ionic radii of RE(III) ions, and the ionic nature of the RE(III)-oxygen bonds, RE(III) ions tend to share the carboxylato-groups to form polymeric complexes. The increase in values due to RE mordanting reveals that dye molecules are capable of forming a metal complex with the positively charged metals as shown in figure-5.

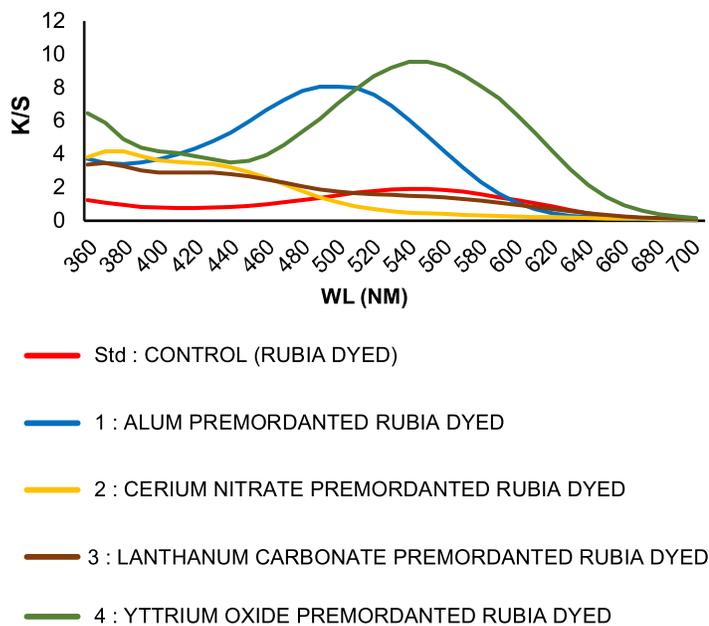


Figure 4 - K/S value graph for different mordants.

The ion of rare earth element can form a bond with hydroxyl and or enone moieties of Natural dye- Rubia dye and the fiber, through their greater coordination complex formation ability, thus rare earth can be aptly used in natural dyeing.

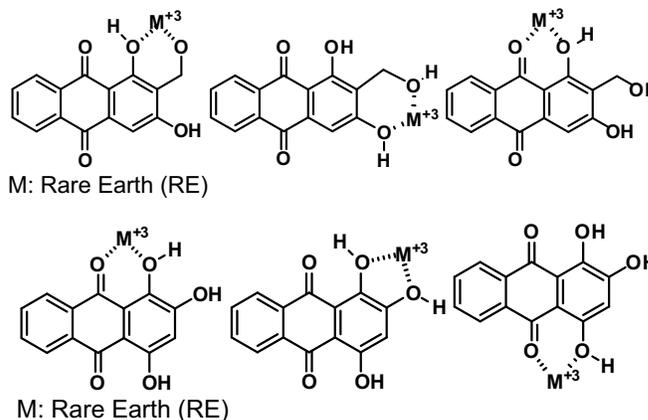


Figure - 5 Metal chelation through oxygen centres

In the case of silk fibres, dye anions and metal cations have a strong attraction towards positively charged amino and negatively charged carboxyl groups, respectively. Hence, they enter the fibre and form ionic bonding between dye and fibre as well as metal ions. The dye-metal chelates thus produced also form coordinate bonds with the uncharged amine (-NH₂) groups of the silk fabric. One molecule of RE mordant can form a bond with one site of fibre molecule. But one molecule of RE mordant can form bonds with several molecules of dyes. As a result, when the mordant molecule binds to fiber it holds many molecules of dye with it.

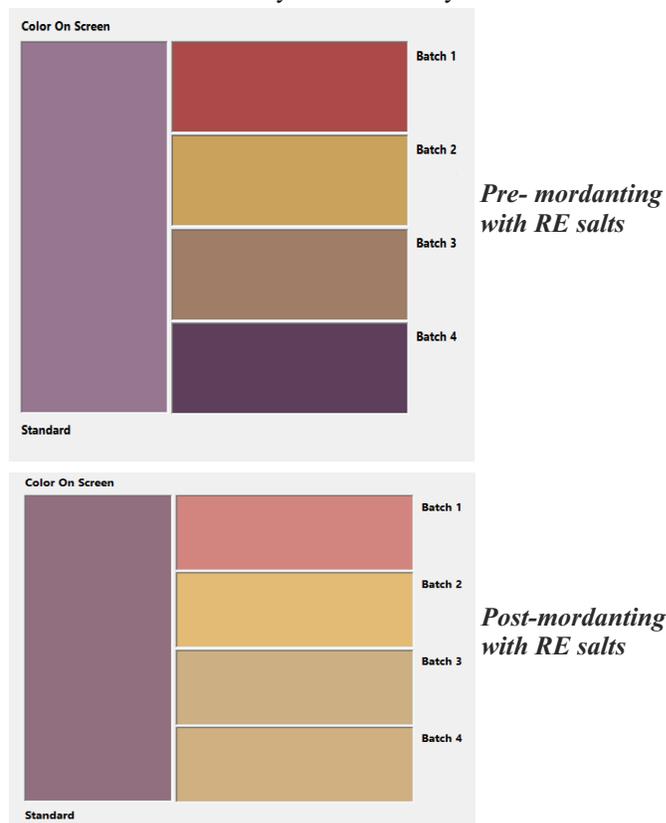


Figure-6 Standard-Unmordanted, Batch-1 Alum mordanted, Batch-2 Cerium nitrate mordanted, Batch-3 Lanthanum carbonate mordanted, Batch-4 Yttrium oxide mordanted.

Therefore, using RE mordant, the colour yield was seen to have enhanced considerably. Thus by the choice of an appropriate RE salt in the premordanting step, we can get different colour from the same dye extract- alum yields orangish red, Cerium salt yields yellowish brown, Lanthanum yields camel brown, and yttrium yields dark purple as shown in figure -5. However, in the post mordanting of the same dye extract, colours obtained on the swatches were very different and were lighter in shade.

The dyeing results of Rubia dye clearly shown in Table-1 and figure-4, clearly indicates that pre-mordanting with yttrium oxide gave K/S value better than that obtained by pre-mordanting with alum. Another observable fact is that premordanting with RE salts is preferred over postmordanting for Rubia dye, considering the CIELab values and K/S values. Out of the three chosen RE salts- Lanthanum carbonate, Cerium nitrate, and Yttrium oxide- the best results were obtained by Yttrium oxide in just 0.4% concentration.

The dyed swatches showed improved wash and light fastnesses as shown in table 2 and the best result was observed in the case of the Yttrium oxide pre-mordanting method. With the use of 0.4% RE salts, the color depth was

found to be very good. Post mordanting did not yield the desired color depth or shades.

4.0 Conclusion:

An intrinsic feature of the lanthanide coordination chemistry is the tendency of the metal ion to maximize its coordination number. Employing rare earth as mordant apparently raised the colour fastness - washing, and light of the silk fabrics dyed with Rubia dye. The study showed that premordanting is preferred over postmordanting for Rubia dye. Out of three RE salts used for premordanting, the best results were obtained for Yttrium oxide. The oxophilicity of Yttrium is owing to the hard Lewis acid character and large ionic radius. RE(III) ions prefer bonding with hard Lewis base donors, such as F, O, and N, and have high coordination numbers. The colorant molecules Manjisthin and purpurin present in Rubia dye have the requisite sites for coordination. By using RE salts in place of alum or other transition metal salts there seems to be manifold advantages- better dye uptake, better fastness properties, low-temperature dyeing, low energy consumption, and above all would have lesser pollution load in the effluent, thus proving them to be ecofriendly and safe mordant.

Table 1 - CIELab values Rubia dyed silk swatches

| S.No | Name | K/S | L* | a* | b* | dE* | Remark |
|---------------|-------------------|---------------|-------|-------|-------|-------|-------------------------|
| Std | Rubia unmordanted | 29.05 | 49.65 | 17.51 | -6.40 | -- | |
| Batch1 | Alum Pre Mord | 99.18 | 40.87 | 46.19 | 16.34 | 37.62 | Change in colour |
| Batch2 | Alum Post Mord | 17.10 | 63.22 | 29.15 | 15.65 | 26.47 | Change in colour |
| Batch3 | CeN Pre Mord | 25.58 | 66.28 | 8.59 | 38.25 | 48.47 | Change in colour |
| Batch4 | CeN Post Mord | 12.06 | 77.84 | 5.95 | 40.71 | 52.93 | Change in colour |
| Batch1 | LaC Pre Mord | 28.83 | 56.75 | 9.30 | 19.93 | 28.48 | Change in colour |
| Batch2 | LaC Post Mord | 10.76 | 72.91 | 4.63 | 25.88 | 38.65 | Change in colour |
| Batch3 | YO Pre Mord | 105.81 | 30.73 | 17.89 | -9.51 | 19.17 | Premordanting/YO |
| Batch4 | YO Post Mord | 11.13 | 73.29 | 5.87 | 27.97 | 40.13 | Change in colour |

CeN- Cerium nitrate, LaC- Lanthanum carbonate, YO- Yttrium oxide, Pre Mord- Premordanting, Post Mord- Postmordanting.

Table 2 - Washing and Lightfastness of the Rubia dyed swatches

| S.No | Name | Washing Fastness | Lightfastness |
|---------------|-------------------|------------------|---------------|
| Std | Rubia unmordanted | 2-3 | 3 |
| Batch1 | Alum Pre Mord | 3-4 | 3 |
| Batch2 | Alum Post Mord | 2-3 | 2 |
| Batch3 | CeN Pre Mord | 3-4 | 3 |
| Batch4 | CeN Post Mord | 3 | 2-3 |
| Batch1 | LaC Pre Mord | 3-4 | 3 |
| Batch2 | LaC Post Mord | 3 | 2-3 |
| Batch3 | YO Pre Mord | 4-5 | 5 |
| Batch4 | YO Post Mord | 4 | 3-4 |

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Installation Damage of Geosynthetics

The geosynthetics are prone to some amount of damage during their installation. To assess the quantity of the installation damage, a standard method was initially developed by Watts and Brady of the Transport Research Laboratory in the United Kingdom. The procedure has also discussed in the ASTM D 5818 with similar requirements. We are at BTRA doing the test following same ASTM D 5818 method followed by respective tensile strength. For the time being we are using the construction site for the sample preparation. If customer will agree, BTRA will collect the sample from site after standard procedure and provide the report.



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Fume Toxicity and Combustion Behavior of Different Polymers and their Health Hazard

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Abstract

The risk of fire is a significant problem in today's society. When a material burns, a number of components are liberated, and poisonous gases are produced as well. Carbon dioxide and carbon monoxide are the two main gases released during a fire, along with hydrochloric acid, nitrogen oxide. Some polymers like polyamides, wool, or silk also release cyanide. These gases can cause a variety of lung diseases, hypoxia, respiratory diseases, fevers, stomach ulcers, and metal diseases due to certain organo-irritants. In this article, we talk about issues relating to the toxicity of textile fumes, such as those from polyester, nylon, aramide fabrics and various polymers like PU, PVC, Fluoropolymer, Styrene etc. Here, the impacts of conditions on the combustion reaction and their burning behavior are described in detail, along with the toxicity of combustion products and assessment of combustion toxicity. The fume toxicity has been studied for different polymers using method NCD 1409.

Keywords

Toxicity, combustion, NCD 1409, PVC, PU

Citation

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1.0 Introduction

One important consideration in the choosing of polymer for diverse applications is the fire risk posed by the materials. Building materials, as well as those used in automobiles, trains, hotels, and home appliances, must meet specific fire performance standards. A flammability test is used to determine a material's flame resistance. The substance degrades thermally in three different ways: through oxidative pyrolysis, anaerobic pyrolysis, and ultimately through flame burning. Below 400 °C, many materials, especially polymers, begin to degrade thermally, releasing poisonous and combustible gases.[2]

Due to their versatility, affordability, and ease of use, polymers are now favored over traditional materials like metals and ceramics in many applications. However, there are concerns due to a larger flammability risk than with traditional materials.[2]

Many polymers have hydrocarbons as their backbones, which makes them more combustible and increases the risk of fire. Polymer shows self-sustaining combustion upon ignition in air. The properties, combustion products, and burning behavior of various polymers vary. According to the material's composition and the fire's temperature, a variety of hazardous chemicals with varying compositions are present in fire smoke.[2]

Ignitability, flammability of volatiles, total heat and its release rate, flame spread, smoke generation, and fume

toxicity are the main hazardous aspects of fire. In the table below, a number of polymers are mentioned along with information about how they are used in most sectors and the gases they emit. (Table 1)

Table 1 - Polymers their applications and combustion product

| Sr. No | Polymers | Applications | Gas evolved during combustion |
|--------|---------------------------|---|--|
| 1 | Polyurethane | Expanded rigid boards, sprayed insulation, flexible foams, elastomers, window treatments, resin flooring, gaskets and thermoplastics rubbers and elastic fabrics. | CO ₂ and CO, HCN, NO and NH ₃ . HCN |
| 2 | Fibre reinforced polymers | Aerospace, marine and automotive industries, furniture, bathroom cabins | CO, CO ₂ and Nox and dangerous inhalable fibers, Particulates |
| 3 | PVC | pipng, window frames, low-voltage cables, insulating sheathing, carpets and PVC coated fabric for seat covering, Artificial wool | CO ₂ , CO and HCL |
| 4 | Fluoropolymers | Wires and cables, insulator coatings of wires | low and unsteady yields of CO ₂ and CO |

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| Sr. No | Polymers | Applications | Gas evolved during combustion |
|--------|-------------|--|--|
| 5 | Polyester | Fabrics (clothing, curtains, and carpets) , bottles and as composites in furniture and appliances | CO, H ₂ O and CO ₂ , benzene, acetaldehyde, formaldehyde and different kinds of hydrocarbons |
| 6 | Polyamide | Flammable interior furnishings and decorative materials particularly textiles), fabrics and consumer and industrial applications. Aliphatic polyamides (nylons) are used in textiles and are important engineering plastics. Aromatic polyamide (aramid) is used mostly in advanced composites | Heavy hydrocarbons, CO, CO ₂ , NH ₃ , HCN and NOx; furans, isocyanates |
| 7 | Polystyrene | As an insulating material, Fire retardant polystyrene foam | Soot, CO, CO ₂ and H ₂ O, aromatic compounds |

2.0 The impact of environmental factors on the start and spread of fire

Major factors impacting fire include temperature, pressure,

water, amount of oxygen present, and ventilation. Evaluation of the combustion products is required since the results of material combustion are greatly influenced by the fire conditions. When a fire starts, the temperature raises, the oxygen level drops, and the concentration of combustion products rises.

Fire fume Toxicity is assessed using a variety of techniques; CO concentration is the primary factor, followed by the development of other harmful gases with lower concentrations. Pyrolysis, charring of the substance and thermal degradation are only a few of the several conditions and stages of fire. First, a solid material is transformed into a gas, and then the material's surface chars, using less energy.

3.0. Health impacts and combustion product analyses.

Only a small portion of fire-related deaths and injuries are caused by the heat and flames; many victims die as a result of the hazardous fire effluents and poor visibility of smoke generation. However, this is dependent on the individual's inhalation rate, exposure period, and proximity to the fire. Because the vast majority of the fire's by products are regarded as toxic.[3]

The Table 2 lists and provides information on the numerous gases that are typically produced during fire incidents, along with any associated health risks.

Table 2 - Combustion product and their effect on human and environment

| Sr. No | Combustion product | Effect on human | Effect on environment |
|--------|--------------------|--|--|
| 1 | Carbon Dioxide | Nausea ,vomiting, rapid breathing, rapid heart rate | The fire effluents may or may not make an impact on environment depending up on the duration of exposure. Effects to the environment are destroying homes, natural habitat and timber, polluting the air with emission of carbon dioxide. Disrupt transporattion, communications, power and water supply, deterioration of the air quality, Release soot particles to the environment. Hazardous chemical are released into the environment through fire plume, figherfighting operations, air and water contamination |
| 2 | Carbon monoxide | Fatigue, headaches confusion and dizziness due to less O2 delivery to brain | |
| 3 | Nitrous Fumes | Breathing problems, Headache, reduced lungs function, Eye Irritation, loss of appetite. bronchitis | |
| 4 | Hydrogen cyanide | Lungs disorder, harmful to blood vessels. | |
| 5 | Ammonia | Burning of the eye, nose, throat and respiratory tract | |
| 6 | Hydrogen chloride | Gastritis, bronchitis, dermatitis .Eyes nose irritation | |
| 7 | Phosgene | Coughing, burning sensation in the throat and eyes difficulty in breathing nausea and vomiting | |
| 8 | Sulfur dioxide | Respiratory tract infection, coughing, asthma. | |
| 9 | Formaldehyde | Burning sensation in the eyes, nose, throat, skin irritation | |
| 10 | Hydrogen sulfite | Dizziness, Headache, Stomach upset, irritation to eye. | |
| 11 | Phenol | Irritation to eyes, nose, throat, skin irritation, muscle ache, skin burn | |
| 12 | Hydrogen fluoride | Body pain, eye irritation | |
| 13 | Acrylonitrile | Weakness, headache, vomiting, dizziness. | |
| 14 | Formaldehyde | Burning of the eye, nose, throat ,coughing and nausea | |

4.0 Analytical approach

By using GCMS, pyrolysis gas chromatography/mass spectrometry (PGC/MS), simultaneous thermal analysis (STA), and pyrolysis-combustion flow calorimetry, it is possible to determine the composition of smoke and other fire products (PCFC).[4]

Small amounts of sample are needed for these procedures. By using infrared polarisation spectroscopy, it is possible to monitor the ongoing generation of HCl during combustion tests (IRPS)[4]

These techniques are effective fire-safe material screening tools for recently synthesized materials. They involve injecting a sample to the GC to separate the mixture's components based on several characteristics, such as boiling point and polarity.[4]

Each developed country has its own set of textile fire testing standard methods which together with those defined by other national and international bodies. ASTM E 662, ISO 5959-2, NF-X 70-100 are different methods for determination of smoke/fume toxicity in US, UK, France etc. In India NCD 1409 is mainly used for the determination of fume toxicity using colorimetric gas detection tubes.

5.0 Test Method:-

The toxicity of fumes is only briefly mentioned in a few mandatory standards. These standards do not include any requirements; instead, they examine the product or material based on its fire qualities.[5]

A minimum amount of fabric or test material is burned for the toxicity index test in a volume of air that produces a maximum toxic concentration of each gas developed during burning. The test technique can be used to specify a raw materials or product's quality because a single test is ineffective for determining the overall fire risk of a product under actual fire situation. This test can be used to compare a variety of synthetic and natural materials' specific combustion characteristics.

The toxicity factor of 14 specific (hazardous) gases produced by complete combustion of the substance in air under a predetermined test environment is added up and assessed using the NCD 1409 test technique. These variables are obtained from the calculated amount of each gas that would be produced when 100g of the substance were burned in air in a volume of 1m³, with the concentration of the resulting gas being expressed as a factor of the concentration fatal to man at 30min. exposure time.[5]

For the detection of harmful gas effluents from fires, colorimetric tubes are usually required. Each gas of interest is captured and analyzed using a separate tube. The majority of the tubes are available in a variety of concentration ranges, and the detectable concentration range may sometimes be altered by the quantity of pump strokes used. Colorimetric tubes are simple to use and can deliver reliable results when used with a single known gas.

Colour change and Cf value for the colorimetric gas detection tube are given in Table 3 below.

The concentration of measured gases is the only parameter used to calculate the toxicity index. Using the provided formula, the Concentration of each gas produced is determined.

$$C_8 = \frac{C \times 100 \times V}{m} \text{ ppm}$$

Where's C=Conc. of gas in test chamber ()

m=Fire test mass (g)

V= Volume of test chamber (m³)

C₈ for all detected gases are used to calculate the Toxicity index.

$$\text{Toxicity Index} = \frac{C_{g1} + C_{g2} + C_{g3} + \dots + C_{gn}}{C_{f1} + C_{f2} + C_{f3} + \dots + C_{fn}}$$

C_f = concentration of gas considered fatal to mass for 30min exposure time (ppm).

C_f values for various gases are given in the standard.[5]

Table 3 - Colour change and Cf value for the colourimetric gas detection tube.[5]

| No. | Name of the gas | Colour change | Value of CF |
|-----|---|--------------------------|-------------|
| 1 | Phosgene (COCl ₂) | White to Red | 25 |
| 2 | Hydrogen Fluoride (HF) | Yellow to Purple | 100 |
| 3 | Hydrogen Chloride (HCl) | Yellow to Pink | 500 |
| 4 | Hydrogen Bromide (HBr), CF=0.8 | Yellow to Purple | 150 |
| 5 | Phenol (C ₆ H ₅ OH) | Yellow to Grey | 250 |
| 6 | Sulphur Dioxide (SO ₂) | Greenish Black to Yellow | 400 |
| 7 | Hydrogen Sulphide (H ₂ S) | White to Brown | 750 |
| 8 | Hydrogen cyanide (HCN) | Yellow to Red | 150 |
| 9 | Acrylonitrile (CH ₂ CHCN) | Yellow to Red | 400 |
| 10 | Formaldehyde(HCHO) | Yellow to Red | 500 |
| 11 | Carbon Monoxide (CO) | White to Brownish Green | 4000 |
| 12 | Carbon Dioxide (CO ₂) - (%) | Blue to off white | 10000 |
| 13 | Nitrous Fumes (NO+NO ₃) | White to Yellow | 250 |
| 14 | Ammonia (NH ₃) | Yellow to Blue | 750 |

6.0 Experimental Data:-

In this paper toxicity studies of PVC coated sheet and the other PU foam, are shown.

6.1.1 In the case of PVC sheet out of 14 hazardous gases as mentioned in the standard, only three gases namely Hydrogen chloride, Carbon monoxide and carbon dioxide are found. The figure 1 shows the colour change in the detection tube.

Table 4 - Toxicity data for the PVC sheet.

| No. | Name of the Gas | Concentration of Gas Generated in ppm (Gas Conc-Blank) | Value of C8 = CX100X0.94/MASS | Toxicity Value of Gas C8/Cf |
|----------------|-----------------------------------|--|-------------------------------|-----------------------------|
| 1 | Hydrogen Chloride (HCl) | 100 | 2346.94 | 4.69 |
| 2 | Carbon Monoxide(CO) | 20 | 469.39 | 0.117 |
| 3 | Carbon Dioxide (CO ₂) | 0.7 | 164286 | 1.64 |
| Toxicity Index | | | | 6.45 |

6.1.2 When PU foam was burned the gases evolved mainly are hydrogen cyanide, carbon monoxide, carbon dioxide, and nitrogen oxides (NOx).The other gases were not detected.

Only the measured gases are used to construct the toxicity index. Results are shown in Table 5; the figure 2 shows the colour change in the detection tube.

Table 5 - Toxicity data for the PU foam.

| No. | Name of the Gas | Concentration of Gas Generated in ppm (Gas Conc-Blank) | Value of C8 = CX100X0.94/MASS | Toxicity Value of Gas C8/Cf |
|----------------|-------------------------------------|--|-------------------------------|-----------------------------|
| 1 | Hydrogen Cyanide (HCN) | 5 | 111.76 | 0.745 |
| 2 | Carbon Monoxide(CO) | 5 | 111.76 | 0.0279 |
| 3 | Carbon Dioxide (CO ₂) | 0.5 | 111766 | 1.11 |
| 4 | Nitrous Fumes (NO+NO ₃) | 1 | 22.35 | 0.089 |
| Toxicity Index | | | | 1.97 |

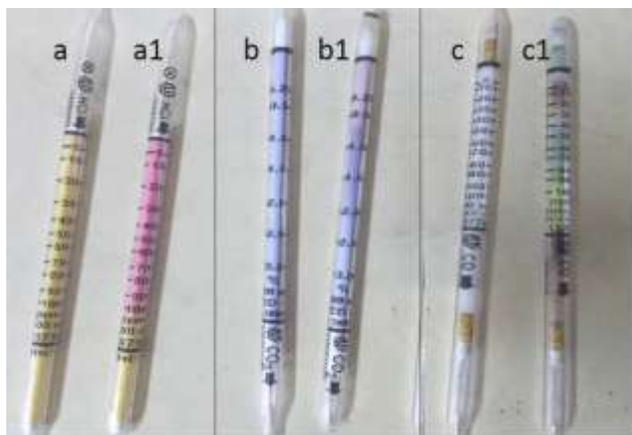


Figure -1:-Change in colour before and after test. Colour change after test is noted by no. 1



Figure-2:-Change in colour before and after test.

7. Conclusion:

The fume toxicity index value of PVC and PU foam is presented in this study. HCL is found in PVC in the higher concentration, followed by CO and CO₂. In case of polyurethane foam in addition to CO and CO₂, HCN and NO_x (NO+NO₂) were also found. For vitiated combustion of N-containing materials, there is often increased generation of

HCN and NO_x. Toxicity index of 1 is acceptable for a given volume, but in the situations mentioned above, the index value is greater than 1, which is caused by the high concentration of HCl and HCN found in PVC and PU respectively. Isocyanates and halogenated gases primarily

arise during the initial stages of the fire and this causes major health hazard to the fire victim.

Therefore, in order to meet the current environmental need, new environmentally friendly flame retardants must be developed in order to replace halogenated ones, isocyanate,

and N-containing polymers.

The further toxicity studies on different polymer will be presented in the upcoming paper.

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New Testing Facilities at High Performance Fibre Facility, BTRA, Mumbai

Dynamic Mechanical Analyzer (DMA) and **Thermo-mechanical Analyzer (TMA)** are very sophisticated type of equipments used to study the mechanical/physical/thermal behavior of a wide range of solid materials subjected to various modes. Both of these equipments are now installed in the "High-Performance Fibre Research Laboratory" at The Bombay Textile Research Association (BTRA). The brief introduction and importance of these equipments in material characterization are stated below:

DMA machine:

DMA machine is used to study the visco-elastic behavior of the polymers under the solid state for the determination of Glass transition temperature, Creep resistance behavior, structural integrity at various temperatures, Stress-relaxation, mechanical behavior, Fatigue behavior on a specific temperature and master curve of polymers in very less time than other conventional mechanical characterization machines. DMA measures these responses in the form of complex modulus (E^*) (total strength of the material), Storage Modulus (E') (Strength of Elastic region), Loss Modulus (E'') (Strength of viscous/rubbery region) and Tan (δ) (ratio of E'' to E') which is extremely difficult to measure by conventional mechanical testing machines/ UTM. The DMA machine installed at BTRA has the capability to study the whole visco-elastic region of polymers and makes it a part of essential equipment required for various applications such as:

1. Tg measurement of polymer, especially pressure-sensitive adhesives and sealants under the pre-loaded condition to simulate a real-application environment.
2. Frequency response on glass-transition of polymer-based diaphragms.
3. Effect of Plasticizer and Filler content on polymer property.
4. Polymer blends compatibility.
5. Characterization of virgin polymer (under powder form) for quality control.
6. Characterization of soft materials and foams.
7. Characterization of single fibre as well as bunch of fibres.
8. Creep behavior and Accelerated Creep behavior (Creep TTS) of Polymers to be used in various applications such as Geo-textile grids, packaging films, conveyors etc.
9. Most importantly the material performance and lifetime prediction using the Time-Temperature Superposition principle.
10. And so on.....



TMA Machine:

Similar to DMA, the TMA facility available at High-Performance Fibre Research Laboratory, BTRA is also capable of studying the thermo-mechanical response of the material in the form of dimensional changes at various temperatures. This equipment gives us an understanding of how a material reacts to its environment which is more important from the application point of view. The TMA facility at BTRA can be used for the various measurements of the materials such as:

1. Determination of coefficient of linear expansion of various materials in the various forms (films, solids, fibres, irregular shape solids etc)
2. Shrinkage/Expansion in the films and fibres.
3. Softening of the material
4. Determination of Glass transition temperature
5. Creep behavior of the material.

And so on.....



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Carbon Fibre - Cost Overview

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Abstract

Carbon fibres are well known for their excellent mechanical property. It's used as a structural material due to its high specific strength in a fibre form supported by its low density compared to conventional metals. Carbon fibre was used in the 19th century & over 70 years of continuous research & development diversified carbon fibre applications from Defense and aerospace to Automobile, wind energy, industrial segments, sports, etc. Its wide usage is limited due to the cost of production. Carbon fibre needs to be cost-effective. For the same, its standard grade's price needs to be reduced from 20 – 25 \$/ Kg to 10 -12 \$/Kg. We herein attempt to provide a summary of a Factor affecting the cost of carbon fibre like Raw material, manufacturing methods, & cost of quality. Here we also discuss the recent cost-effective manufacturing technologies like Cut carbon fibre (CCF) for composite-based applications, Textile grade PAN & advanced processing techniques like plasma oxidation for low-cost Carbon fibre.

Keywords

Carbon fibre, Precursors - Polyacrylonitrile, Pitch, Renewable resources – Lignin, Textile grade PAN (T – PAN), Advance plasma oxidation techniques, Cut carbon fibre (CCF), Tow size, Cost of the quality, Cost reduction, Application.

Citation

Prashant Gharal - Carbon fibre and its cost, *BTRA Scan* - Vol. LI No. 3 July 2022, Page no. 15 to 19

1.0 Introduction

Carbon fibre is a superior material known for its versatile properties like mechanical strength (tenacity 3-6 GPa, modulus of 300- 500 Gpa), low density (1.8-2.2g/cm²), and excellent formability into different composite products [1]. It is used in aerospace, high-performance cars, equipment for defence, and windmill application [2,4]. More than 96% of commercially available carbon fibre is based on Polyacrylonitrile (PAN) based chemistry, which gives the best product at a commercially viable price [3]. The standard grade carbon fibre, which has the most significant share of commercially produced fibre with average tenacity in the range of 3.5GPa, is sold approximately in the price range of USD 20-22\$ [5]. Some of the carbon fibres used for aerospace applications with higher modulus or tensile strength are sold even at a higher price than this. The cost of carbon fibre became the main hurdle in its growth.

Recent governmental, environmental, and industrial consortium efforts are pushing the automobile industry to reduce its carbon footprint [6, 8]. One way to reduce carbon emissions and increase the fuel efficiency of an automobile is to mitigate its weight [9, 10]. Here the low-density (~1.8 g/cc) carbon fibre with moderate tenacity of 1.75 GPa and modulus of 175GPa can become a solution [11, 12]. The industry desired the price of carbon fibre to be below US10\$/kg to make it commercially viable [13]. It led to a lot of research on low-cost carbon fibre. Research studies are

coming up the different materials and improved processes to reduce the cost of carbon fibre [14-19]. Textile grade carbon fibre is gaining much attention in this regard; ORNL has developed a new method to process the textile grade carbon fibre and get the desired quality of carbon fibre with the targeted price [20, 21].

2.0 The inception of carbon fibre manufacturing:

To understand the current manufacturing scenario of carbon fibre, we need to understand the inception of carbon fibre manufacturing. Carbon fibre as a material has been well known since the late 19th century, as Mr. Joseph swan and later Mr. Thomas Edison developed them for electric bulb application from cellulose. But it was in 1958 Dr. Roger bacon at Union carbide's R&D that developed a graphite whisker. The short carbon fibre has a strength of 20 GPa and modulus of 700 GPa, which created a new interest in carbon fibre manufacturing [22]. Union carbide later patented the longer carbon fibre manufacturing by graphitisation of rayon fibre [23].

Rayon is regenerated cellulose and carbon fibre produced from this process, giving a low process yield (less than 30%). Meanwhile, around the same time, Polyacrylonitrile as acrylic fibre for textile application was also developed at the commercial level at Dupont and sold under the brand name Orlon. After world war-II at Government funded institute GIRIO in Japan, Dr. Akio Shindo developed a process for producing the carbon fibre from Polyacrylonitrile using an

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intermediate slow oxidation process which gave a material yield of 50-60% [22]. Toray Industries developed this process further and since then led as the biggest manufacturer of the carbon fibre industry. In England, during a similar time, Courtauld developed a special acrylic fibre (SAF) with itaconic acid as a co-monomer for acrylic fibre dye-uptake. SAF had the drawback that it was getting yellow during the processing stage. Later they used this SAF for a faster oxidation process in the carbon fibre process. Even though PAN-based carbon fibre gives very high tensile strength, the overall process yield (~50%) and multiple manufacturing stages kept the cost of the product very high. Another material used as a carbon fibre precursor was Pitch. Pitch is an anisotropic composition of aromatic hydrocarbon. In 1970, Union carbide also developed mesophase pitch-based carbon fibres. The mesophase pitch has a good process product yield of ~80% and gives less load on the abatement process. The carbon fibre produced has a tensile modulus of ~900GPa. But the tenacity is less compared to PAN-based carbon fibre.

Earlier applications of carbon fibres were primarily related to defence applications like shells for missiles or as aeroplane components. Later, in 1972 the American golfer Jim flood won the golf tournament using a carbon fibre shaft-based golf club. It started the use of carbon fibre in sports gear applications. Post-cold war, the Demand for military applications was suddenly reduced, which led to a change in the focus of research on the utility of carbon fibre. With constant research and incremental development in the carbon fibre industry over 70 years, its usage diversified in different fields like wind energy, aerospace, automobile, and industrial segment.

Besides the technical challenges to manufacturing carbon fibre, its manufacturing cost becomes its biggest hindrance. Compared to a standard grade low carbon steel (1.1\$/kg) or aerospace-grade aluminium(2.5\$/kg), a carbon fibre cost is several times higher (range of 20 to 25\$/kg). Despite its superior mechanical property, low density, and corrosion resistance.; its usage and production are limited. So current research work is mainly focused on producing low-cost carbon fibre.

Recently published [24] information about carbon fibre manufacturing and its demands in composite applications indicates the healthy growth in the carbon fibre market. In the year 2019, 161200 tonnes of carbon fibre were produced. The Japanese company “Toray” is aggressively pursuing growth, and they have produced 57000 tonnes of carbon fibre which is almost 35% of the world's total production. They recently took over leading composite manufacturer “Tencate”, showing their commitment and ensuring the market for their produced fibre. After Japan, which has 43% of total world manufacturing capacity, country-wise, the United States ranks second with 25% of world production, mainly contributed by Hexcel and Mitsubishi Corporation. Chinese, Taiwanese, and SouthKorean are also trying to

increase their manufacturing capacity. Chinese top five manufacturers having less than 10000 tonnes capacity per year contribute almost 13% of the global carbon fibre production. Kangdexin (in Changing, china) is said to have an aggressive plan to expand its manufacturing capacity to 70K tonnes per year.

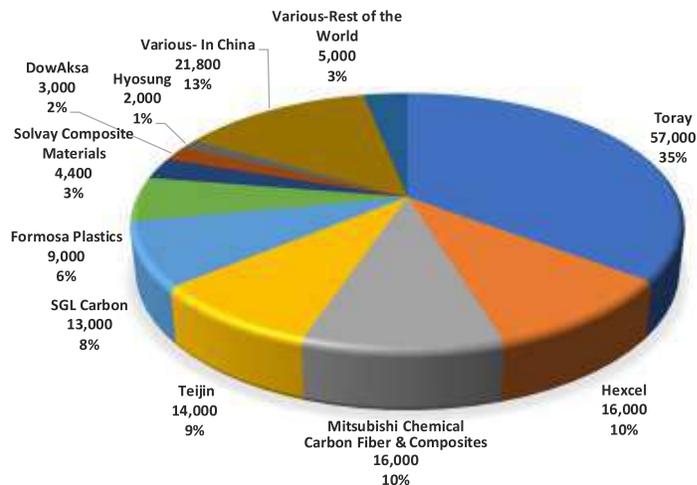


Figure 1 - Carbon fibre manufacturing capacity - year 2019

The carbon fibre industry has product varieties according to their end-use. They can be classified as per their mechanical strength or their tow size, which is the number of filaments in a bunch. Tow sizes are designed as per the requirement of the end application and the manufacturing processes of composite products. But broadly, we can classify them as per the end application like Aerospace, Industrial, and sports/leisure, which are broadly defined by the different strength grades of carbon fibre.

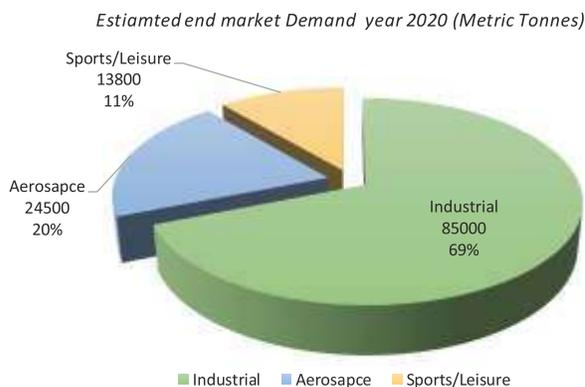


Figure 2 - Demand for carbon fibre as per application sector - year 2019

The overall yearly demand for carbon fibres as per the estimation until 2025 will reach the value of 191350 MT compared to today's 123300 MT. Steady growth in the industrial sector where Demand will reach for wind energy (27,300 MT), transportation (22,750 MT), infrastructure (20,800 MT), and pressure vessels (19,500 MT). The aerospace sector will require 30000MT, of which 15500MT

will be used in commercial aeroplanes. The sports and leisure market will also show growth and Demand of 19000MT.

The following table gives an idea about carbon fibre's classification based on the end application, its required mechanical strength, and the Tow size used. Based on Tow size (i.e. number of filaments/bunch), they can be classified as a Small tow (≥ 24000 or 24k filaments) which will be sharing 70% of the overall carbon fibre demand compared to large tow (≤ 24000 filaments like 40k or 50k) (Das et al. 2016). High modulus fibres (more than ~ 500 GPa) are primarily used in aerospace and defence applications, where their cost is affordable in terms of end-use. Standard modulus grade fibre has almost 80% market share today. The standard modulus or general-purpose grade is sold approximately at 20\$/kg.

| Type | Modulus (GPa) | Strength [MPa] | Tow Size (K) | Application |
|----------------------|---------------|----------------|--------------|--|
| Standard Modulus | 230 | 3,500 | 12–50 | Automotive, Aerospace |
| Intermediate Modulus | 400 | 5,000 | 3–24 | Pressure Vessels, Wind Turbine Blades, Aerospace |
| High Modulus | 500 | 3,500 | 1–12 | Aerospace |

Table 1 - Grades of carbon fibre as per application sector (Das et.al)

But in the current situation of the COVID-19 pandemic, [25], these projections will be changed or delayed in the years to come. Post-pandemic, the world order will be changed. The way we were doing the business will change. Hostilities within countries will change trade relations. With economic recessions, the industry will be under tremendous pressure to sustain manufacturing and maintain the product cost. The aviation industry is severely affected by the current situation, and it will take a longer time to revive and get back into the business. Naturally, which will put a hold on aeroplane manufacturing and, subsequently, demand aerospace-grade carbon fibre, one of the costliest grades in carbon fibres.

Another factor that is ignored most of the time is the cost of quality. It's nothing but a loss in profit due to poor product quality [26]. It is known that the carbon fibre industry runs or control all the process step with tight tolerance. Ideally, the process tolerance coefficient of variation on percentage yield is targeted below 1.0%. But it is observed in real life that for small tow (1K to 24K), it is in the range of 3%, and in the case of large tow (more than 24k), the tolerance limit is in the range of 15%. Based on the tolerance limits on product quality, there will be a production of sub-standard products and production waste, which will increase the cost of

manufacturing or reduce the profits. Due to the secretive nature of the carbon fibre production process, it is challenging to get the actual data on carbon fibre's cost and the impact of various process controls on the product quality. The cost of the quality and the size of fibre production are also interlinked. The increase in two sizes in the same production facility increases the overall production capacity.

This commercial carbon fibre precursor material, i.e. PAN, Pitch, and Rayon, has advantages and drawbacks in terms of fibre properties and processability. Overall the carbon fibre with desired physical properties is costlier.

3.0 Carbon fibre its end application in composites:

At the initial stage of development, the Demand for carbon fibre was limited. They were mainly used for defence applications. In 1960, American golfers demonstrated the superiority of carbon fibre-based golf-club compared to conventional construction materials. It opened a new area in sports goods. Japan's Toray took the lead in this area and significantly developed the carbon fibre variety for sports applications.

Carbon fibre growth is always going hand-hand with growth and development in composite manufacturing. The cost of the Composite product defines the Demand for carbon fibre. Composite manufacturing defines the required properties of carbon fibre like tensile properties, surface treatment, and tow size. E.g. For aerospace applications, high modulus (500GPa) and high strength (more than 3.5Gpa) carbon fibre with a tow size of lesser than 12k filaments are preferred. Large tows (24k to 50k) having standard (230Gpa) or intermediate (400Gpa) modulus; are used in automotive parts or windmill application [3].

The automotive market is very price sensitive. As per Auto-industry, for successful commercial utilisation of carbon fibre, its cost should be 10-12\$/kg range, Which is driving the development of low-cost raw materials for carbon fibre manufacturing. A low-cost renewable resource like lignin is one of the candidates in the development process.

Composite manufacturing processes like filament winding, autoclave moulding, compression, and resin transfer moulding; have their issue. Like longer manufacturing time, resin curing time, energy-intensive autoclave for curing, labour intensive prepreg layup process which adds up to the final cost of the product [27]. New development in Cut Carbon fibre (CCF) from large tow carbon fibre is promising for the automotive application [28]. CCF is compatible with most composite-making processes like stir and slip casting, compression moulding, and automated spray deposition. It overcomes the disadvantages of long carbon fibre processing, where it requires unique processing like filament winding, co-curing, and tow spreading, which significantly increases its processing cost. So, it is evident that the demand for carbon fibre in industrial application development in low-cost composite manufacturing is essential [29].

4.0 Carbon fibre manufacturing in terms of its raw material and end product cost:

Carbon fibre production was first started with rayon based process, but due to lower product yield (33%) and low mechanical strength, it is losing its competition with PAN-based processes. The use of Polyacrylonitrile as the prime material for manufacturing carbon fibre compared to other raw materials is the ease of production with acceptable quality fibres and high product yield. Nowadays, there is a strong drive towards using cheap renewable resources like lignin as raw material, but it's still in the research and development stage.

Carbon fibre cost depends upon the raw material and production process. We can classify carbon fibre manufacturing in broadly three steps. Raw material preparation for precursor, precursor spinning, and precursor thermal treatment. A precursor is a raw material in the fibre form that will be converted into carbon fibre by thermal treatment.

Following are the various raw materials available and used to manufacture carbon fibre. The base raw materials are petroleum-based products like Polyacrylonitrile, Pitch, or natural sources like regenerated cellulose rayon. Precursor formation involves two steps that are raw material preparation and fibre formation by spinning process. Precursor thermal treatment is divided broadly into two stages, i.e. stabilisation (oxidation) and carbonisation. There is significant material loss during thermal treatment due to pyrolysis, which gives a negative product yield. Product yield has a direct and major impact on the cost of final carbon fibre.

| Precursor | Cost (\$/kg) | % yield | Cost of carbon fibre (\$/kg) |
|-------------------|--------------|---------|------------------------------|
| PAN | 3.0-6.0 | 50-55 | 20-25 |
| Pitch | 1.0-2.0 | 80-90 | ~60.0 |
| Rayon | ~2.2 | 10-30 | NA |
| Melt spun PAN | 3.15 | =50 | 13.0-17.0 |
| Lignin | ~1.52 | 30-55 | 4.0-6.27 |
| Polyethylene | 1.57-2.36 | 70 | NA |
| Textile grade PAN | 2.2-2.5 | ~50 | 11-12 |

NA-not available

Table 2 - Costs of carbon fibre based on precursor yield

Table 1 indicates the cost of precursors produced from different raw material sources. The cost of the carbon fibre was derived based on the %carbon yield and the approximate precursor thermal processing cost. The values indicated lags the detailed costing as per the Tow size and processing conditions; it is given as a general idea to understand the product yield and cost. PAN chemistry-based precursors which are produced by the wet spinning method, are

available at the price of 3.0-6.0\$/kg. Considering 50% carbon yield, it turns out to be a costlier precursor. Large tow size carbon fibres produced from PAN chemistry like SGL's sigrafil C (24k-50K tow size) and Zoltek's Panex-33(48K tow size) are sold at the cost range of 15.4-24.2\$/kg.

Pitch-based carbon fibres are costlier even though Pitch is a cheaper petroleum by-product. Standard grade carbon fibre from Pitch needs to be converted into mesophase Pitch for manufacturing. This conversion process of the isotropic Pitch to mesophase is costly. It requires careful chemical processing to get the desired level of high molecular weight polyaromatic structures like asphaltenes.

The melt spinning method reduces the cost of spinning due to higher spinning speed (3 times the wet spinning), and it also avoids the usage of hazardous solvents [30,31]. To make it melt process-able, PAN polymer can be co-polymerised with higher co-monomer content (10% methyl acrylate), which negatively affects the overall carbon yield of the final carbon fibre [32]. Alternatively, plasticizers (like water) are also used to ease melt processing. However, it requires a specially designed melt processing system to handle the high water pressure [33, 34]. The carbon fibres produced also have micro-voids (act as weak spots), which leads to lower tensile strengths (~2.5GPa). Overall, this method is still in the concept stage, and the properties of resultant carbon fibres are inferior to wet spun PAN-based carbon fibres. Pitch is a cheaper petroleum side product [35]. The cost of purification of Pitch and converting it into mesophase (required for high-strength fibre) escalates the cost of pitch precursor higher than the PAN precursor [36]. Rayon is not the preferred precursor as the %carbon yield is very low [37].

To lower the cost of precursor polymer, various precursor chemistries like lignin and polyethylene were explored. Lignin precursor is one of the cheapest precursors available at the cost of ~1.5\$/kg [14, 39, 39]. Lignin goes through complex chemical reactions during stabilisation. It gives a lower material yield (10-28%) with low strength carbon fibre (tensile strength 1.2-2.5GPa). Sulphonated polyethylene is considered for melt spinning application which gives carbon fibre with better carbon yield of (>60%) and medium tensile strength in the range of 2.5GPa [40, 41]. Commercially this process is not viable due to the handling of corrosive sulphonation reaction. Efforts were made to use textile-grade PAN as a precursor for cost-effective carbon fibre. However, the absence of essential carboxyl functional co-monomer, low polymer molecular weight (≤ 105 Da), higher precursor fibre denier, low molecular orientation, and low tensile strength of precursor fibre resulted in low tensile strength carbon fibre [42, 43].

Another approach for reducing the cost of the precursor is blending PAN with low-cost lignin. Carbon fibres produced by blending had a micro-void issue on the fibre surface and had a low tensile strength of 1.2GPa. Compared to other precursors' chemistry, PAN-based precursors are the costlier but superior option in manufacturing and have better tensile properties [44].

The lower cost of textile grade PAN compared to PAN precursor may be attributed to the scale of manufacturing, i.e. producing 100K filament size tow compared to less than 50k filament size tow [29]. Textile grade PAN is processed using an advanced plasma oxidation technique that has been demonstrated at a laboratory scale. SGL/FISIPe, in collaboration with ORNL, has developed carbon fibre from textile grade PAN-based copolymer of vinyl acetate and methyl-acrylate. At the experimental level, they demonstrated carbon fibres with desired tenacity. They expect the cost of carbon fibre produced using textile grade

PAN and advanced processing techniques like plasma oxidation will be at ~11-12\$/kg [20, 21].

Looking at available raw material options, textile grade PAN seems to be a viable option to produce in the existing manufacturing setup. At this point, we want to understand the key differentiators between the Commercial PAN precursor and textile grade PAN (T-PAN) fibre in terms of its physical and chemical properties and need to highlight the process and hardware changes required to use T-PAN as a carbon fibre precursor

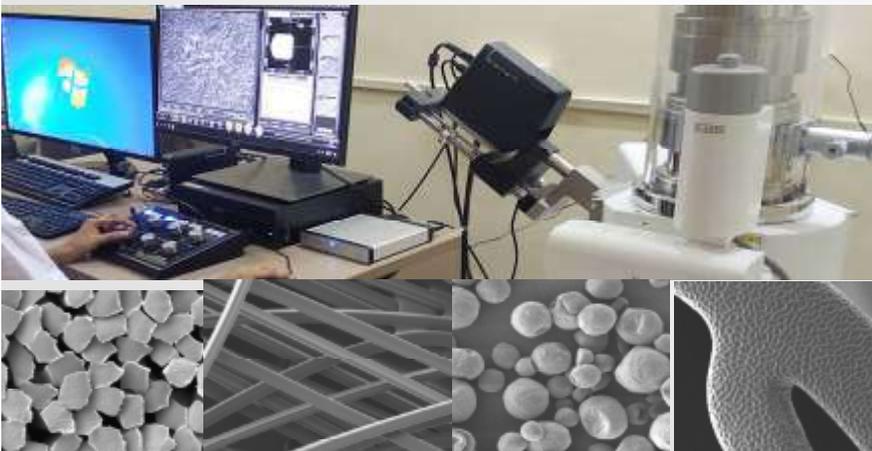
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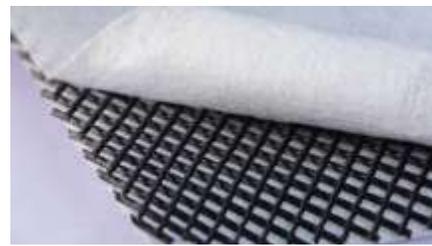
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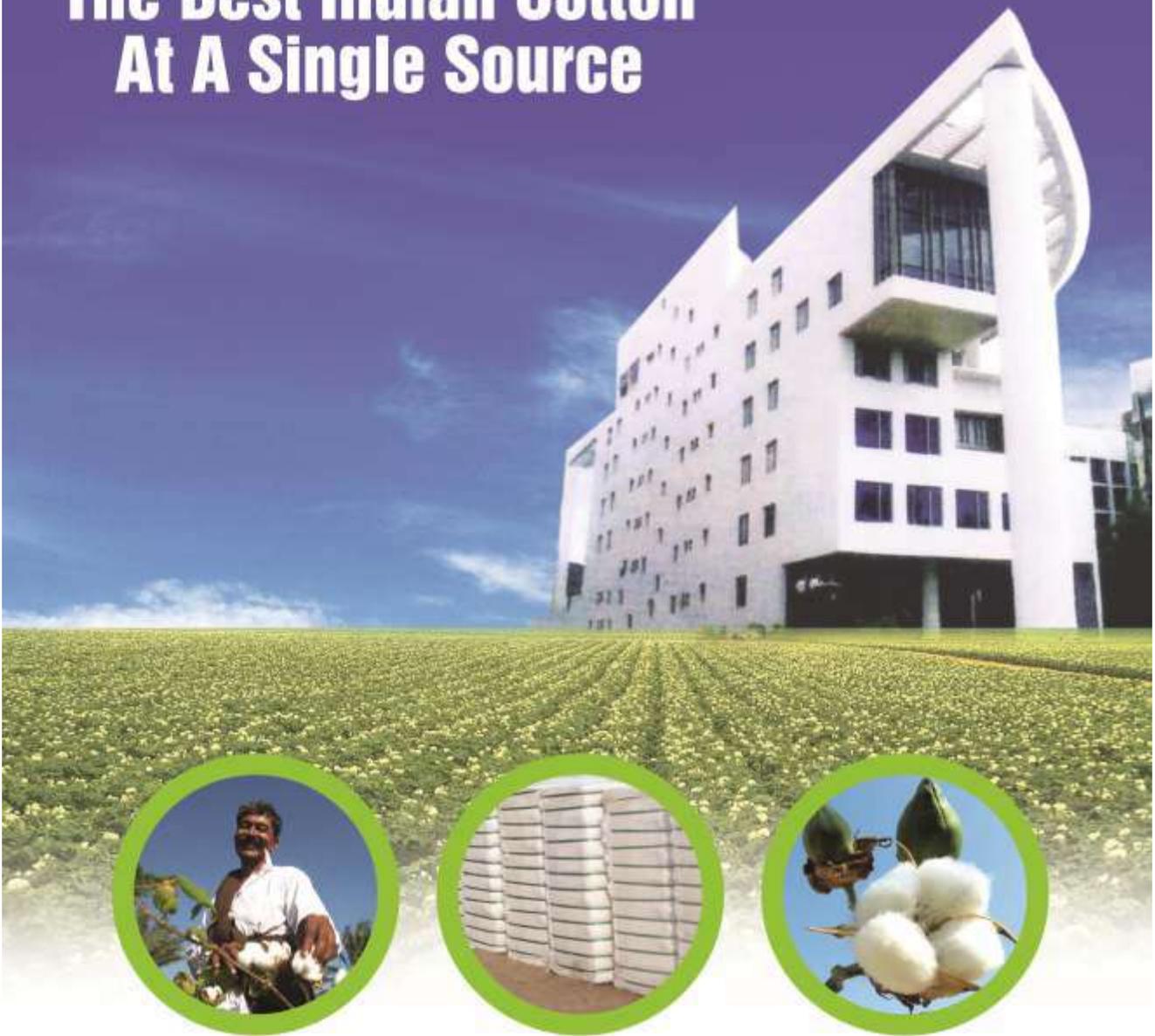
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