MASS COLORATION OF POLYESTER FIBER – EMERGING OPPORTUNITIES FOR GREEN SUSTAINABLE DYEING TECHNOLOGY

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Abstract:
Polyester, as a synthetic fiber, exhibits remarkable versatility and has gained significant acceptance and utilization in the textile industry since its inception. It currently holds the prominent position as the dominant fiber in clothing production. However, polyester fiber faces certain limitations when it comes to dyeing, primarily due to the high glass transition temperature and absence of reactive groups. Earlier the carrier agents were employed during dyeing but it had detrimental environmental effects. Now a days, polyester fibers is being dyed using high-temperature and high-pressure processes with disperse dyes. To overcome these challenges, alternative dyeing methods for polyester fiber have been sought.

One such technique is Mass Coloration or may be termed as Spin dyeing, where a color masterbatch is incorporated into the molten polymer during the melt-spinning process prior to fiber formation. This innovative approach eliminates the need of water resources and significantly enhances the environmental sustainability of the product.

The present paper provides a comprehensive discussion on both Mass Coloration and Conventional Dyeing techniques, including their respective advantages and disadvantages. Additionally, technical and industrial aspects related to the coloration of polyester fiber via Mass coloration are thoroughly examined.

Keywords: Eco-friendly dyeing, Exhaust HTHP dyeing, Polyester fiber, Pre-mix, Spin dyed or doped dyed.

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1.0 INTRODUCTION

Polyester polymer was first developed in 1939 by Whinfield and Dickson. Later, by 1941, a patent for the synthesis of Polyethylene Terephthalate (PET) was granted to the two chemists. With support of C.G. Ritche and W.K. Birthwistle, polyester was spun into fiber for the first time in the same year. This new fiber got the brand name TERYLENE® and was developed for ICI (Imperial Chemical Industries). In 1950, the patent and all rights were sold to DuPONT®, who marketed this polymer later under the brand name DACRON®. Finally, in 1951, polyester was introduced to the public for the first time.

Polyester is one of most valuable member of synthetic polymers due to its versatile applicability for their excellent thermal and durable physical characteristics [1-3]. PET macromolecules are highly compact in nature and exhibits high melting point and glass transition temperature [1]. Polyester-fiber possesses extraordinary robustness against thermal cycling, sun exposure, moisture and measureable chemical degradation/ reactivity [1].

Polyester contributes about 18% of world polymer production and is the fourth-most-produced polymer after polyethylene (PE), polypropylene (PP) and polyvinyl chloride (PVC).

According to the Textile Value Chain report, India recorded a substantial export of US$ 4.7 billion worth of apparel made from Man-Made Fiber (MMF) in 2021. Over the period from 2010 to 2021, the exports of MMF-based textile and apparel have demonstrated remarkable growth, escalating from US$ 6.8 billion to US$ 11.6 billion, representing a 5% increase. Notably, there was a significant surge in the export of technical textiles, apparel, and home textiles, showing growth rates of 13%, 8%, and 6%, respectively. However, exports of fabrics experienced a slight decline of 2% during the same period. Analyzing the Indian MMF textile and apparel exports, it is evident that apparel holds the largest market share, accounting for 41%, closely followed by technical textiles and fabrics, both with a 14% share each.

India must enhance its market share in the synthetic fabric and apparel trade by prioritizing the manufacturing of highly traded and rapidly growing fabric and garment categories. To achieve this goal, the Indian government is actively endorsing the growth of the Man-Made Fiber (MMF) sector within the country. Various policies have been introduced to transform India into a prominent hub for synthetic textile manufacturing. These policies include the Production Linked Incentive (PLI), Mega Investment Textile Parks (MITRA), and National Technical Textiles Mission (NTTM). The implementation of these initiatives aims to strengthen the synthetic fabric and apparel industry in India and boost its competitive edge in the global market.

India's installed polyester capacity is experiencing substantial growth. Presently, the country possesses an installed polyester capacity exceeding 7000 KTPA (Thousand Tons Per Annum), and it is projected to witness an annual growth rate of 3.3% (Compound Annual Growth Rate - CAGR). Within the next three years, this capacity is expected to reach a staggering 8245 million tons. This significant expansion in polyester capacity reflects India's increasing focus on the textile industry and its position as a key player in the global market.

2.0 GLOBAL MARKET SCENARIO OF POLYESTER FIBER

Since its conception, polyester is being considered as promising candidate in textile industry [1]. The global fiber market is predominantly composed of synthetic fibers, which make up approximately two-thirds of the market [18], while the remaining one-third consists of various natural fibers based on proteins, such as cotton and wool, as well as man-made fibers derived from regenerated cellulose, such as acetate or viscose. Of all the synthetic fibers, polyester is the most widely used and holds the largest share of the global fiber market [19]. The sky rated profile of polyester materials demand and production Figure 1(a) is clear evidence of its huge share and decisive role in the global market [2].

For over three decades, the demand for polyester fibers has been growing at an unprecedented rate. As of 2018, the PET market was valued at 100 billion USD and is projected to grow at a CAGR of more than 8% between 2019 and 2026. (**Figure 1(b-c)**).

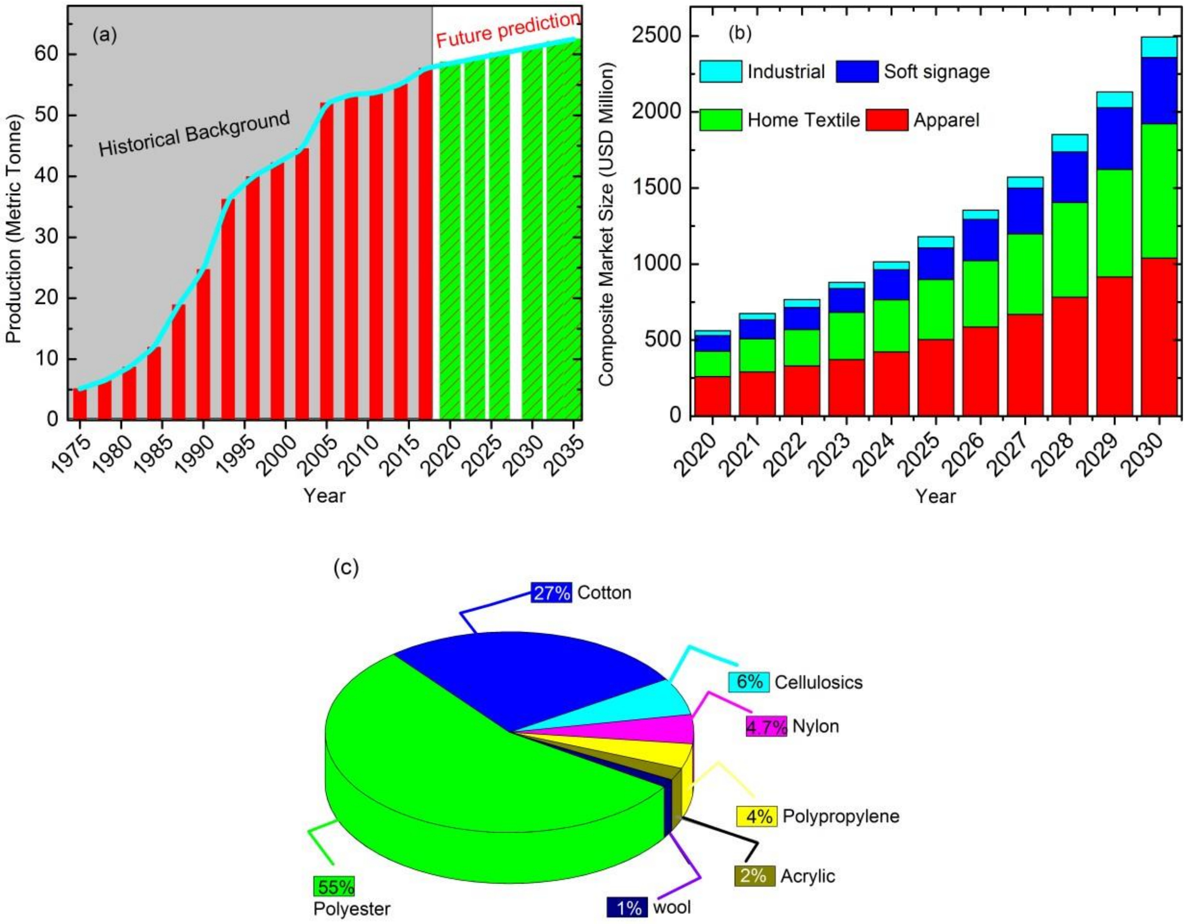

Figure 1 (a) - Production of synthetic fibre. (b) Composite market size of polyester with 15.9% U.S. Market CAGR. (c) Market share of primary fibres.

3.0 DOWNSIDE OF THE POLYESTER FIBERS

Despite of dominating feature of Polyester fibers there are certain limitation like lacking of active polar centres that ensure hydrophobic characteristics of polyesters-fibres, as a consequence, the absorption of water molecules in polymer system is quite hard and enables water resistance and swelling proof characteristics [1]. The hydrophobic nature of polyester fibers limits the ability of dye molecules to spread throughout the fiber. As a result of poor lateral movement of the dye molecules, the process of colouring is not very effective, and achieving an even distribution of color throughout the fiber can be quite challenging. This emphasize that coloration should be done under specialized physical protocol of high temperature and high pressure. In addition, dispersed dye particles of fine size and of non-ionic in nature substantially catalyses the colouring behaviour of synthetic fibres. Moreover, the detailed morphological analysis reveals that surface structure of fibre remain stable against the high temperature and pressure treatment. The scientific finding reported in literature insights that dyeing of polyester fibre with solution without association of metallic mordant is quite promising approach.

This allows realizing technologically anticipated colouring performance of synthetic fibres and in pursuit of eco-friendly dyeing mechanism to eliminate the footprint on environment.

4.0 CONVENTIONAL DYEING OF POLYESTER FIBER - HTHP EXHAUST DYEING PROCESS

Today, the dyeing of raw polyester material with a dye dissolved in an aqueous medium is a well-explored and widely established technique in the textile industry. This process allows the fiber to be dyed in various forms, such as fiber, yarn, fabric, or slivers. Over the years, this dyeing method has undergone several technical advancements, including the implementation of new dyes, chemicals, and state-of-the-art synthesis and characterization techniques. The initial step involves dyeing the polyester fibers, resulting in a grey (uncolored) fiber or cloth. The grey fiber is then immersed in a dye solution, and the dye molecules are absorbed, facilitating the transfer of color from the liquid (aqueous) phase to the solid organic substance (fiber).

To achieve dispersion dyeing, disperse dyes are mixed with water and surface-active chemicals to form an aqueous dispersed solution, which is then transferred to a coloring vessel. The colored aqueous solution is optimized to have a fiber/liquor ratio of about 1:20 to ensure proper soaking of the fibers and consistent coloring. This ratio also ensures uniform coloration of the substrate polymer.

However, dispersed dyes have limited solubility in the aqueous medium. The aqueous solution contains a balance between dissolved and undisclosed dye substances. While the dissolved dye actively contributes to coloration due to its strong affinity towards the fiber, the undisclosed dyes come into play during the coloration process, improving its efficiency. The color molecules deposited on the fiber substrate gradually diffuse into its interior.

For optimal results, the coloration process is carried out under high temperature and high-pressure conditions. Elevating the temperature enhances the energy of the dye chemical, while the high pressure further promotes penetration of dye molecules by causing swelling of the fibers. This swelling facilitates homogeneous coloration, and the dye molecules become tightly bound to the fiber due to numerous hydrogen bonds and Van der Waals forces.

In general, a typical dyeing process (Batch Dyeing) using disperses dyes is characterized by following process steps which are carried out subsequently:

Stir the dye into the aqueous phase. Depending on process, this can happen at elevated temperatures and can also involve the addition of dispersing agents or other auxiliaries which accelerate the entire dyeing process.

- pH is measured. For optimized conditions, the value is adjusted by addition of acetic acid.
- Soak the polyester material in this dyeing bath.
- Keep it for 15 minutes at a temperature of 60°C.
- The temperature of the dye bath is subsequently raised to 130°C and held at this level for approximately 1 hour.

During this period, the dye undergoes partial dissolution in the dye bath and gets adsorbed on the surface of the fiber. The rate at which the dye molecules move to the fiber surface is influenced by their affinity to the substrate and their concentration in the bath.

- The process involves the adsorption of dye from the fiber surface into the core of the fiber substrate. The desired outcome is a fiber cross-section that is uniformly dyed, representing optimal coloration. It's important to note that, typically, the adsorption rate surpasses the diffusion rate under normal conditions, making it the controlling step in the dyeing process.
- As final steps, the dyeing batch is cooled down. The dyed material is rinsed with water and - if required - a reductive cleaning step is carried out. Eventually, the dyed material is rinsed and dried.

For visualization of the dyeing process (**Figure 2**), please refer to the following scheme:

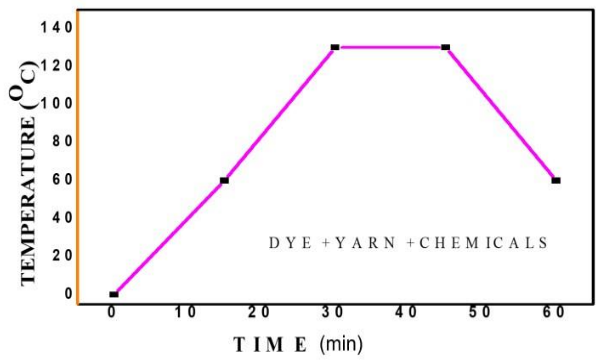

Figure 2: Dyeing batch process

5.0 ENVIRONMENTAL IMPACT OF CONVENTIONAL DYEING

In the industry, the inhomogeneous cross-sectional coloration of polyester filaments and expensive coloration processing limits the potential application in textile fabrication [1,2]. The conventional coloration process involves excessive use of chemical additives and auxiliaries along with dispersed phase of fine colorant residue [1]. The chemical activity of colorants, at ambient temperature and pressure, degrades with addition of chemical auxiliaries during the dyeing process [1]. Most importantly, the conventional coloration route requires huge proportion of water, become polluted and cannot be recycled even after rigorous treatment, and enormous waste of electrical energy, in order to follow standard protocol of coloration e.g. high temperature 130 °C and high pressure. This widely accepted coloration technique exhibit a hazardous footprint on environment [1]. During the dyeing routine, huge quantities of waste water are discharged, generally contains significant amount of dyes as well as auxiliary constituents used for solution standardization [1]. The level of contamination is still high even after several treatments. For the safe and pleasant stay of our coming generation, it is our duty to quest an alternative for the existing conventional technology of coloration. Of course, the conventional routes are being regularly revisited and a amendments are included time to time to improve the coloration process. From economic and eco-friendly point of view, numerous significant advancement in coloration technique etc. has been achieved via lowering the excessive use of raw chemicals and reusages of waste water after rigorous insitu treatment [1].

6.0 MASS COLORATION OF POLYESTER

In the field of synthetic fibre coloration, presently, mass coloration technique is well appreciated for their excellent performance, most economic approach and zero wastage of life-saving-water [1]. In this technique, the colorants are added into the molten polymer, as intermediate step, in form of a solid/ powder or paste, before the commencement of fibre formation. There are different types of color concentrates available. Beside their physical appearance - solid or liquid - it can also differentiate by following virtues:

- Mono-Concentrates which are formulated with one colorant only
- Tailor-made-Master batches, which are based on a formulation containing more than one colorant in order to match a required shade requested by a customer.

6.1 ADVANTAGE OF MASS COLORATION TECHNIQUE OF POLYESTER FIBER

Mass coloration, spin dyeing or dope dyeing is a cost-effective method of coloring used for large-scale applications such as the automotive industry [17]. The advantages of mass dyeing over traditional dyeing methods are:

- Excellent fastness properties
- High production rates resulting in significant savings in utilities, energy, and manpower services
- Zero water pollution
- Lower coloring costs
- Ability to texture, colored yarns without negatively affecting the shade [1,2]

There are several mass coloration techniques that are being employed towards the production of colored polyester at large scale [3]. In this article, we briefly emphasize on the two leading mass coloration techniques (though direct melt spinning) in technical and industrial perspective of polyester coloration (through CP route).

6.2 PRODUCTION OF MASTER BATCH

As mentioned above, solid Master Batches can typically be used for the mass coloration of polyester fiber. When applying this coloration route, the preparation of master batch plays a very important role since the quality of the color concentrate is key to success. The Masterbatch is heavily loaded with Polymer soluble dyes, organic and inorganic pigments so mainly the quality of Masterbatch depends on the dispersion of colorants in the carrier resin. For getting better dispersion the mixing of ingredients in high speed mixer, Screw profile of twin extruder and process parameters has to be optimized.

Following, typical process steps for the production of a solid masterbatch (granules). This process can be applied for pigment based concentrates as well as for dye based ones.

6.2.1 Preparation of pre-mix: Colorant (pigment or polymer soluble dye) and carrier polymer are precisely weighed separately. For the production of pigment based concentrates, wetting agents are added; for special end use, also specific additives such as UVA's, HALS or optical brightener can be added.

6.2.2 Mixing of Ingredients: All the Ingredients used are fed in parts one after other in the high-speed mixer to prepare homogenous mixture. The mixer started at low, medium and high speed and time is controlled by the set temperature.

6.2.3 Extrusion: After cooling at room temperature the Pre-mixed batch is collected and fed into the hopper of a Twin-ScrewExtruder. Alternatively, also a split-feed process, in which polymer, colorants or additives are fed at any point between the feeding zone and the outlet of the extruder into the molten polymer stream, can be applied. The appropriate placement of conveying, grinding and mixing elements in screw profile are very important factors to deciding the quality of masterbatch,

6.2.4 Pelletizing: The molten colored polymer mass leaves the die head of the extruder and is cooled in a water bath. The polymer strands are then cut into granules using a pelletizer.

6.2.5 Packing: The granules are bagged or sent to the spinning department after being sieved to remove dust.

Twin-Screw Extruders are frequently used for the extrusion process. The majority of extruders used for master batching contain intermeshing, co-rotating screws set on splined shafts in a closed barrel to provide highly flexible conveyance, compression mixing, shearing, and heating. In screw profile 35 to 45% mixing and kneading eliments are kept and balance is for conveying the molten material.

The mechanical shear from the screw and the heat from the barrel, convert the solid polymer into a molten mass. This mass is then pressed through the die, is cooled in a water bath and cut into granules. This technology is widely established and provides a high efficient homogenization.

For the masterbatches, an appropriate carrier polymer has to be chosen. The majority of high quality concentrates are based on Polybutylene Terephthalate (PBT) resin. For standard qualities also Polyethylene Terephthalate (PET) can be used. Although more costly, PBT is the preferred substrate because of thermal stability, its lower melting point and the lower sensitivity to hydrolytic degradation[1]. As a colorant for master batches, pigments as well as polymer soluble dyes are used. This class of dyes in powder form is residue-free soluble in molten polymer. Pigments may be of organic or inorganic nature. Both have in common that they are not going into solution in hot molten polymer mass. In contrast to pigments, polymer dyes are soluble; at least in limited concentrations.

In order to get the most out of coloration with pigments, they have to be well dispersed in the carrier polymer. This guarantees that the concentrates develop the required shade - hue and especially color strength, but perfect dispersion also provides trouble free down-stream processing. Depending on the chemistry of reactants, surface activity and crystal form, pigments more or less tend to agglomerate or form aggregates; inorganic pigments to a lesser extent than organic ones. To break these agglomerates into smaller particles during extrusion, processing conditions like feeding rate, addition of lubricants or wetting agents, screw speed, throughput and temperature during extrusion have to be optimised. The dye based master batches are usually produced in concentrations between 25% and 40%. In case of pigments, the colorant loading of such concentrates is typically not more than 30%, for inorganic ones also higher.

Typical dosing of master batch to polymer is between 1 to 5% depending on concentration of master batch and the requirement of particular shades. Other than master batches, we find also suitable compounds in the market. They are solid master batches which contain a much lower concentration of colorant since they are not intended to be dosed into the polymer, they are used without additional processing. Compounds are mainly used in the plastic industry.

7.0 QUALITY ASSURANCE OF MASTER BATCHES

Various properties are defining the quality of color concentrates. Following characterization techniques plays an important role and may be used as decisive criteria:

7.1 Filter Pressure Value (FPV): To estimate the homogeneity of distributed colorants in the masterbatch, the FPV is determined. This is the simply done by taking a defined amount of concentrate, mix it with a fixed amount of carrier polymer and fed this mix into the characterizing equipment. This test material is extruded and forced through a test screen. The resulting pressure rise is measured and reported in unit of bar/g.

7.2 Shade Matching**:** shade matching is done by spinning a given dosing ratio of master batch into filaments on a pilot spinning machine. The shade then is compared by use of a spectrophotometer against the standard shade.

7.3 Fastness properties: Service properties such as color fastness to light, washing or sublimation, are tested on knitted fabric samples. To ensure that there is no color bleeding during down-stream processing; fastness to washing is checked at 90°C for duration of 30 minutes with 1% nonionic detergent solution.

7.4 Thermal stability: the thermal stability of a master batch is checked as weight loss by use thermo gravimetric analysis (TGA). Test parameter: 290°C during 30 minutes.

7.5 Granulometry: Shape and size distribution of granules. Also the granule weight as chips/g is checked.

8.1 YARN/ FIBER SPINNING VIA MASTERBATCH ROUTE

To perform mass coloration, i.e., the color in the form of Master batches is mixed with polymer just before melt-spinning process; before the fiber is formed. The masterbatch is dried at 100 to 120 degC and fed into the extruder mouth by gravimetric K-Tron feeder. Doing so, all down-stream process steps for dyeing become obsolete. Other than mentioned in fig. 3a & b, alternatively also a side-stream extruder can be installed. In this smaller and simpler extruder, further, master batch is melted and then this mass is fed into the molten virgin polymer stream of the main extruder. In order to homogenized the material, static mixing elements in the main extruder or a dynamic mixer at the outlet of the main extruder is installed.

Today, spinning pumps with integrated dynamic mixers are also available. Such pumps can replace the conventional spinning pumps at the outlet of the spinning extruder and additional increase the level of homogenization.

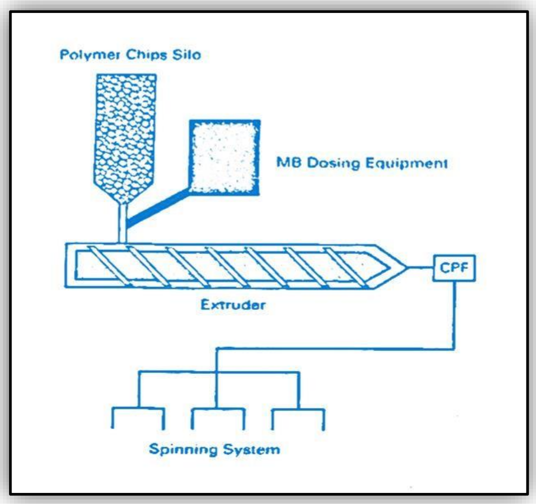

Figure 3a: Joint-Feed Process

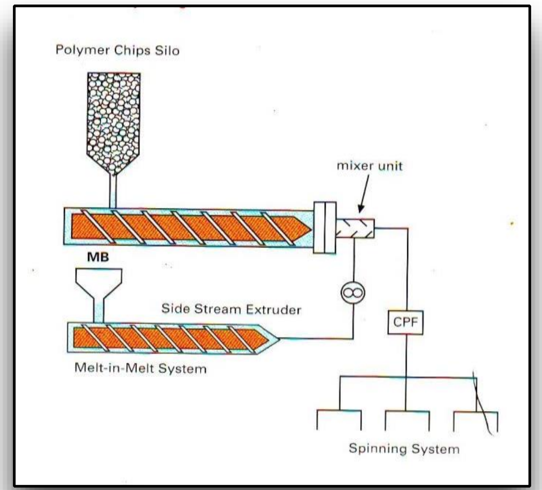

Figure 3b: Injection Process

Figure 4: Process Steps – Polyester spin Dyeing (Mass coloration)

8.2 CONVENTIONAL DYEING VS. MASS COLORATION

Coloration routes, conventional dyeing and mass coloration, are presently in active use. Thus, for lucid representation and better understanding and make a real distinction between the two techniques, it will be appropriate to make a comparative listing of the advantages and disadvantages of the two processes.

Conventional Dyeing

- Suitable economic route for small production lots
- Solution for a wide variety of textile applications
- Exclusive coloration route for space dyeing

- Not cost effective for small production volumes
- Lot-to-Lot variations
- Time consuming process
- Difficult to adopt for flat yarns
- High energy and water consumption
- Generation of wastewater and large quantity of effluents
- High operational and labor costs
- Complex process and recurring cost

Mass Coloration

- Integrated production and huge time savings
- Consistant product quality
- Superior luster and haptic
- High color fastness to light, washing and other service properties
- Works with all filament- and fiber-qualities and cross-sections
- Much 'greener' process. No waste water, low energy consumption
- Lower investments and lower labor cost.

- Not cost effective in lot sizes of less than one ton
- Restrictions in production of colored fiber blends

The technologies that leave no environmental footprint are highly valued in the current, rising economic and technical environment [22]. The textile industry is currently thought to be the one that releases the most pollutants globally. Leading federal institutions and the premier government have voiced their concerns and asked for action to address the environmental deformation. The textile industry is, however, paying close attention to the issue of pollution and attempting to introduce intelligent coloration techniques for polyester fibre. According to recent studies, textile waste takes up over 5% of total landfill space. Additionally, textile processing and dyeing are responsible for more than 20% of all fresh water contamination.

Statistically, polyester dyeing is a major cause of fresh water pollution with conventional high-pressure, high-temperature mechanism being the primary route used for coloration of polyester. Not only does this process consume a high amount of water but there are serious issues related to waste water treatment. Unfortunately, most of these dyes can bypass waste water treatment and persist in the environment as a result of their good fastness to UV and their resistance against temperature, chemicals, soap and even bleach. In addition, additives like anti-microbial agents used for the finishing of fibers are a problem in waste water treatment since they hinder the bacteria to clean the water by biodegradation. Therefore, there is certainly a need for new and more effective processes. Which require low water consumption and reduced color loading for better waste water treatment. Mass Coloration provides an alternative that doesn't require water for the coloration of fibres and significantly reduces the environmental hazards, thus, mitigating problems related to coloring of polyester.

Conclusions

Polyester fibre can be dyed in two ways: conventional HTHP exhaust dyeing and mass coloration. The colour is added to the dye bath in conventional dyeing, which is the classic procedure, and the dyeing is done at high temperature and high pressure for around 60 - 90 minutes. The method produces a wide spectrum of colours with good brilliancy. However, there are issues with uniformity when larger quantities are required, as well as limitations regarding fastness or insufficient penetration in the case of certain materials. However, the primary concern is environmental.

It necessitates a large consumption of water and electrical energy, as well as the creation of dirty water and other difficult-tomanage byproducts. Mass coloration, on the other hand, employs dye or pigment concentrates that are placed directly into the molten polymer prior to fibre production during the melt-spinning process. It fully eliminates the need of water, lowering the product's water footprint. As a result, because of its inherent advantages and good features, we will see more and more mass coloured polyester fibre in the future. And it is unquestionably the more intelligent technique to colour polyester fibre.

Declaration of competing interest

The authors hereby declare that they have no known competing financial interests or personal relationships that may have influenced the research work presented in this review paper. This statement is being made with the intention of providing full transparency and accountability to the readers of this paper.

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Recent Highlight* -- JK Srivastava attending ITMA 2023, Milan, Italy. On 10th June 2023, during ITMA 2023, SDC chief executive officer Dr Graham Clayton (standing left) presented an FSDC (Chartered Colourist, Fellow of the SDC) certificate to Mr Jitendra Kumar Srivastava, head of the Technical Excellence Centre for fibres at Coraplast. Coraplast is a subsidiary of Colourtex Group and a prominent manufacturer of masterbatches, emphasised in its expertise in producing custom colours and additives for the Dope Dyeing process. Its specialist offerings cater to applications in PET, PBT, PA6, and PP, with a particular focus on key industries such as automotive, carpet manufacturing, bath rugs, and apparel production. The presentation took place on the Colourtex stand, which was focused on sustainable innovation and the company's complete line of dyestuffs applicable to both textile and nontextile applications. Showcased was an exclusive collection of colorants, designed specifically for supercritical CO2 dyeing of polyester, a range of digital printing inks comprising disperse dyes for sublimation printing, and reactive and acid dye inks. Additionally, water-based pigment preparations for textile printing, and an environmentally friendly range of modified basic dyes, were highlighted.

ITMA continues to be a hub for gathering valuable market insight, as well as acknowledging the innovations and technologies that continue to push the industry forward. This year's conference placed a predominant focus on advanced materials, pioneering technologies, automation and the principles of sustainability and circularity. *SDC's blog Post (https://sdc.org.uk/sdcs-post-show-updates-from-itma-2023/) SDC or The Society of Dyers & Colourists is a charity registered in England & Wales, plus a Royal Chartered body.