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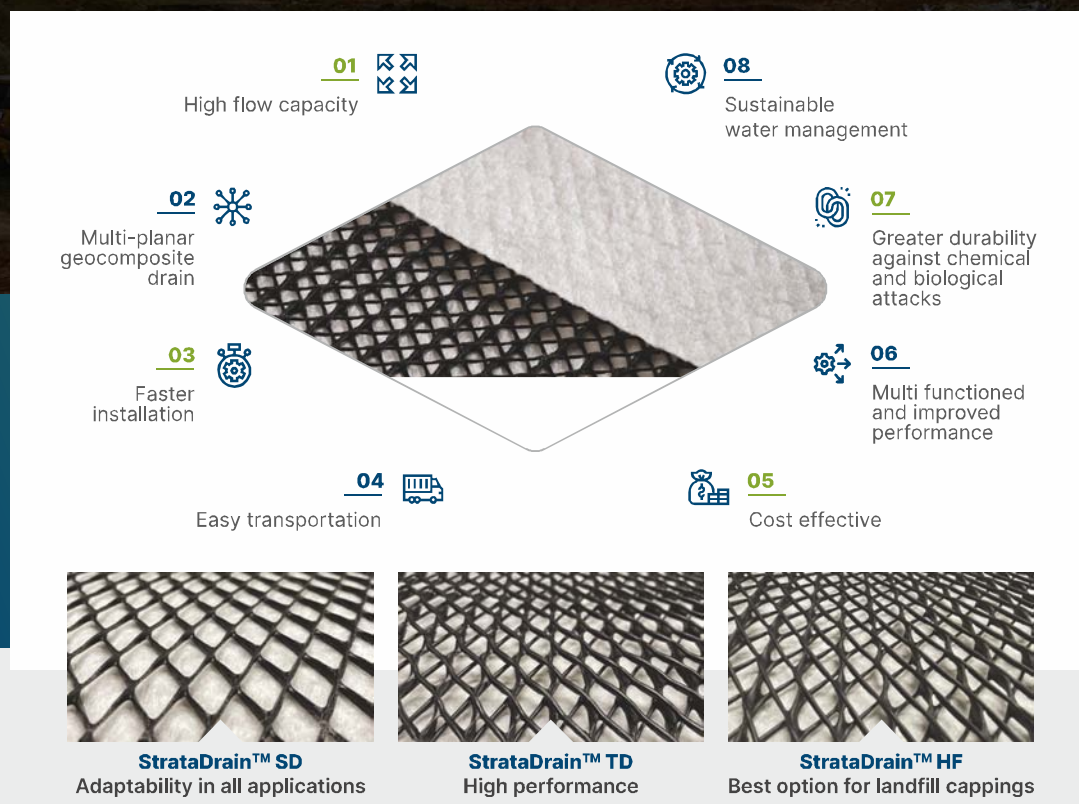


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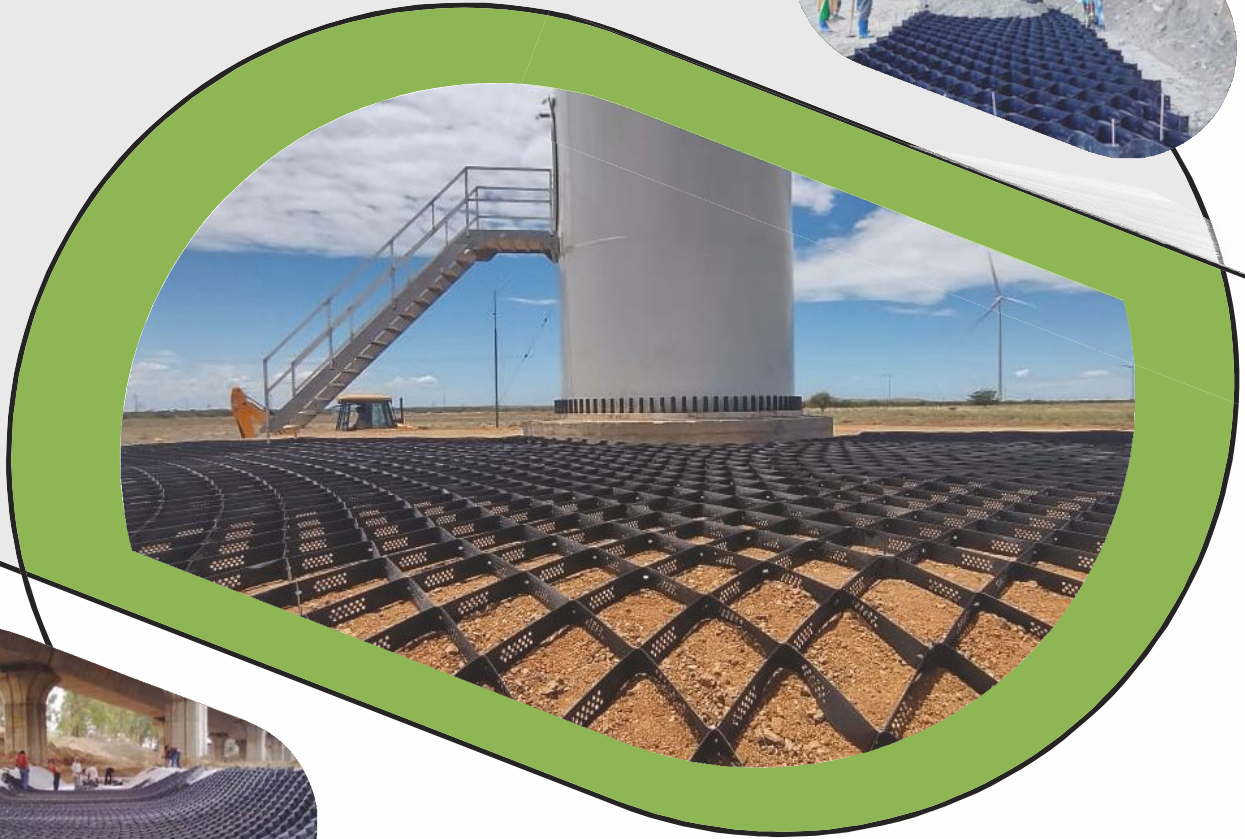
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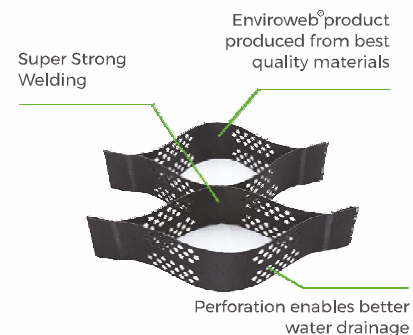
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EDITOR'S DESK

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. We are working 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 4th issue of 55th Edition of BTRA SCAN.

This issue has 3 papers from the different domains such as Comparative Analytical Characterization and Chromatographic Profiling of Virgin and Recycled Polyethylene Terephthalate (PET), Developments of standard procedure to identify dope dyed and exhaust dyed meta aramid yarn and Transitioning to Green Textile Manufacturing: The Synergistic Role of Microbial Enzymes and Biosurfactants. 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 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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Comparative Analytical Characterization and Chromatographic Profiling of Virgin and Recycled Polyethylene Terephthalate (PET)

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Abstract

Polyethylene terephthalate (PET) and its copolymeric variants represent a critical class of thermoplastic polymers widely deployed in packaging, industrial textiles, and consumer products due to their tensile strength, dimensional stability, and chemical resistance. PET materials are broadly categorized into virgin polyester (vPET), derived directly from petrochemical feedstocks, and recycled polyester (rPET), produced via the mechanical or chemical reprocessing of post-consumer polymer waste. While functional performance across both forms remains comparable, structural differences in chain morphology, thermal degradation history, and comonomer distribution persist. In this study, gas chromatographic (GC) profiling following depolymerization, extraction, and derivatization was evaluated as a qualitative and quantitative diagnostic methodology to differentiate vPET and rPET matrices based on residual monomeric markers. Method validation according to IS 16481-2016 demonstrated successful detection of isophthalic acid (IPA) across five commercial food-contact plastic substrates. Chromatographic comparison revealed distinct profile signatures, wherein rPET exhibited a secondary retention peak ($t_R=8.565$ min) attributable to residual monomeric IPA, whereas vPET displayed a singular internal standard peak ($t_R=6.386$ min). These findings demonstrate the viability of chromatographic profiling for verification, quality assurance, and traceability of recycled polymer fractions within commercial supply chains. The article aims to improve awareness of the origin and applications of polyester fibres and to highlight how recycling existing polyester waste can contribute to resource conservation and waste reduction.

Keywords

Polyethylene terephthalate (PET); Isophthalic acid (IPA); Gas chromatography; Textile fibres; Awareness; Sustainability.

Citation

Tanvi Patil*, Shraddha Nitturkar, Geetanjali Mishra, Karishma Hemani, "Comparative Analytical Characterization and Chromatographic Profiling of Virgin and Recycled Polyethylene Terephthalate (PET)", *BTRA Scan* - Vol. LV No.4, Oct., 2026, Page no. 5 to 10, DOI: 10.70225/006648iitgkt

1.0 Introduction:

Polyethylene terephthalate (PET), commonly known as polyester is an aromatic semi-crystalline polyester widely synthesized via the polycondensation of terephthalic acid (TPA) or dimethyl terephthalate (DMT) with ethylene glycol (EG) (given in Fig. 1). Due to its high barrier efficiency against gases, structural rigidity, and optical clarity, PET constitutes a major fraction of post-consumer packaging materials and synthetic textile fibers. Virgin PET (vPET) is synthesized through direct petrochemical routes, producing

highly uniform macromolecular chains characterized by low polydispersity, high intrinsic viscosity, and minimal contamination. Conversely, recycled PET (rPET) is derived from post-consumer streams (e.g., mineral water bottles, packaging films, and discarded textiles) via mechanical sorting, shredding, thermal extrusion, or chemical depolymerization (1). Visual representation of Virgin PET and recycled PET is given in Fig. 2. While advanced processing can yield rPET with mechanical properties approaching those of vPET, repeated mechanical recycling inevitably induces chain scission, thermal-oxidative degradation, and hydrolytic cleavage.

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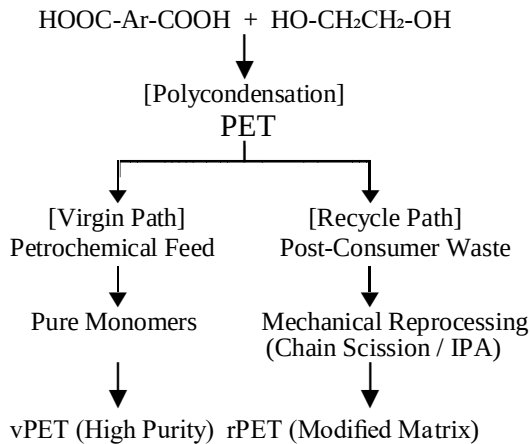


Fig. 1: Synthesis process of PET

Key characteristics of virgin polyester are;

- **Uniformity & purity:** Highly consistent molecular weight distribution with zero contamination or structural variation.
- **High tenacity:** Superior tensile strength, elasticity, and dimensional stability due to unbroken polymer chains.
- **Color clarity:** Pure white optical properties that easily absorb light, bright dyes without color contamination.
- **Environmental impact:** High carbon footprint, intensive energy consumption, and reliance on non-renewable fossil fuel resources.



Fig. 2: Virgin and recycled polyester fiber

Uses of virgin polyester includes activewear, home textiles like curtains, baby wipes, bedcovers, pillows etc. The schematic presentation is depicted in Fig. 3.



Fig. 3: Schematic representation of uses of virgin polyester

At the same time, recycled polyester is manufactured by reprocessing the existing post-consumer PET waste (such as clear water bottles, packaging materials, or discarded textiles) into new usable fibers (1). The key characteristics of this are;

- **Performance parity:** Modern processing methods yield fibers that closely match virgin polyester in strength, durability, and texture.
- **Slight thermal/chain variations:** In mechanical rPET, slight thermal degradation can occur over repeated cycles, though chemical recycling restores virgin-equivalent purity.
- **Dyeing & dye Affinity:** Trace comonomers (such as isophthalic acid from original PET bottles) alter polymer crystallinity, which often improves dye uptake at lower temperatures.
- **Environmental Impact:**
 - Diverts plastic waste from landfills and ocean ecosystems.
 - Requires significantly less energy and generates fewer carbon emissions than virgin petro-chemical extraction.



Fig. 4: Schematic representation of uses of recycled polyester

The flowchart given in Fig. 5 outlines the step-by-step mechanical recycling process used to transform post-consumer PET bottle waste into high-quality recycled polyester fibers.

To optimize the crystallization kinetics, thermal stability, and stretch-blow molding behavior of bottle-grade PET, comonomers such as isophthalic acid (IPA; benzene-1,3-dicarboxylic acid, $\text{C}_6\text{H}_4\text{O}_4$, structure given in Fig. 6) are intentionally incorporated into the polymer backbone at fractions typically ranging between 1 to 5 wt%. The asymmetric meta-substitution of isophthalic acid disrupts chain regularity, lowering the rate of crystallization and increasing the amorphous fraction of the polymer matrix. This structural alteration enhances optical transparency in rigid packaging and alters dye susceptibility in textile fibers by increasing dye diffusion into amorphous domains.

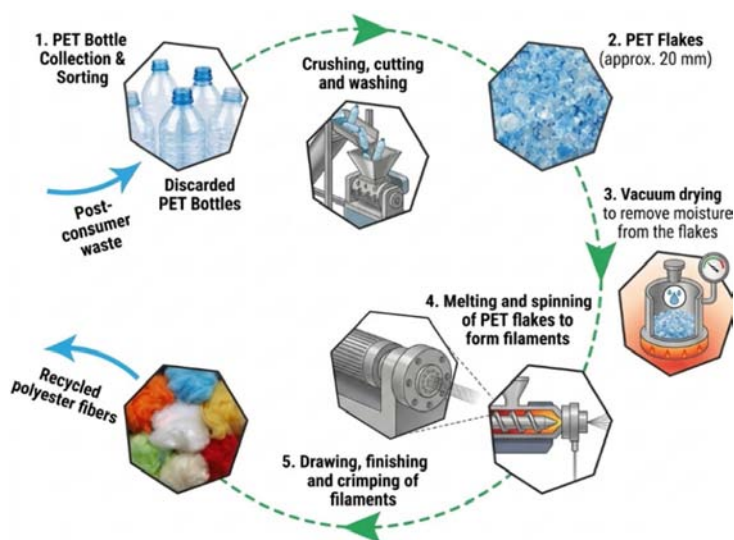


Fig. 5: Flow-chart showing the recycling process of PET bottles

During polycondensation, thermal processing, or subsequent service life, incomplete conversion or hydrolytic cleavage can yield unbound residual monomers and oligomers within the polymer matrix. Identifying and quantifying these unreacted or liberated small molecules is critical for quality control, material traceability, and evaluating mass migration into contacting media.

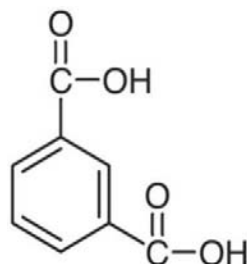


Fig. 6: Structure of isophthalic acid

Ideally, all monomers become chemically integrated into the polymer backbone during polymerization. However, trace levels of residual monomers may remain in the finished product due to incomplete conversion, manufacturing conditions, thermal degradation, or hydrolytic degradation during service. Monitoring residual aromatic acids in food-contact polymers is therefore important for product quality, manufacturing consistency, and migration studies (2, 3).

This study demonstrates a gas chromatographic screening methodology following chemical cleavage and derivatization to quantify residual IPA content in commercial food-contact packaging material and evaluate its utility as a diagnostic marker for differentiating vPET and rPET matrices. The method is intended for rapid screening and routine laboratory analysis. This analysis can create awareness among common public to minimize the use of recycled polyester as it contains IPA which is carcinogenic in

nature and continuous exposure if it to food, skin can cause cancer and other health related issues.

2. Experimental

2.1. Reagents and Materials

Chromatographic grade benzyl alcohol, titanium tetraisopropoxide ($\text{Ti}(\text{OCH}(\text{CH}_3)_2)_4$), and chloroform (CHCl_3) were obtained from commercial suppliers and used without further purification. Commercial post-consumer polymer samples representing direct food-contact packaging applications were acquired from local commercial suppliers:

- Sample-A: Fruit juice containers (PET)
- Sample-B: Mineral water packaging (PET)
- Sample-C: Disposable cutlery (Spoons)
- Sample-D: Rigid food storage containers
- Sample-E: Transparent thermoformed food packaging

2.2. Sample Preparation and Depolymerization

The polymer samples were rinsed with ultrapure water, dried under vacuum, and mechanically comminuted to a uniform particle size of approximately 3-5 mm. Quantitative samples weighing 0.20–1.00 g were transferred into sealed reaction vessels. Chemical cleavage and extraction of the bound and unreacted isophthalic acid fractions were conducted in accordance with the standard IS 16481-2016. To render the non-volatile dicarboxylic acid suitable for gas chromatographic separation, the extracted analytes were subjected to esterification/derivatization to yield volatile dialkyl esters prior to injection.

2.3. Instrumental Analysis

Qualitative and quantitative evaluations were conducted using a Gas Chromatograph equipped with a Flame Ionization Detector (GC-FID) / Total Ion Chromatogram (TIC) acquisition setup.

- Injection Volume: 1.0 μL
- Separation Profile: Isothermal / program-optimized to achieve baseline resolution of derivatized IPA from background matrix components.
- Quantification: Standard addition and peak area integration methods according to IS 16481-2016 parameters.

3. Results and Discussion

3.1. Quantitative Determination of IPA Content

The developed method produced well-resolved chromatographic peaks for the derivatized IPA with minimal interference from polymer additives or extraction solvents. Adequate chromatographic separation was achieved within a relatively short analytical run, allowing efficient routine

sample through put. Good linearity was observed over the investigated concentration range, demonstrating the suitability of the method for quantitative analysis. Repeat injections produced reproducible peak areas and retention times, indicating satisfactory instrumental precision and stable chromatographic performance. Sample preparation was a critical step in the analysis because direct chromatographic determination of the target acid is limited by its physicochemical properties. Therefore, hydrolysis followed by derivatization was employed to convert the analyte into a more suitable volatile derivative for GC analysis (4-6). The derivatization step improved chromatographic response and peak symmetry, thereby facilitating reliable detection and quantification. The determined IPA contents of the investigated packaging materials are presented in Table 1. IPA was detected in all five samples, with concentrations ranging from 1.78% to 2.10%. Sample-B and Sample-C showed the highest measured concentration (2.10%), followed by Sample-A and Sample-D (2.00%), while Sample-E showed the lowest concentration (1.78%). The mean IPA content across the five investigated samples was approximately 2.00%.

Table-1: IPA content of different packaging materials

Samples	IPA content (%)
Sample-A	2.0
Sample-B	2.1
Sample-C	2.1
Sample-D	2.0
Sample-E	1.78

The corresponding chromatogram is given in Fig. 6 (virgin polyester) & Fig. 7 (recycled polyester). The relatively narrow range of measured values in Table-1 indicates that IPA was present at comparable levels across the investigated packaging materials. The observed variation may be associated with differences in polymer formulation, manufacturing conditions, raw-material composition and processing history.

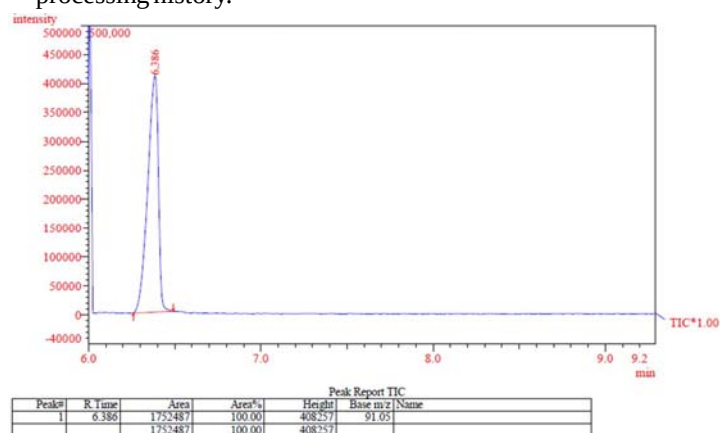


Fig. 6: Chromatogram of virgin polymer

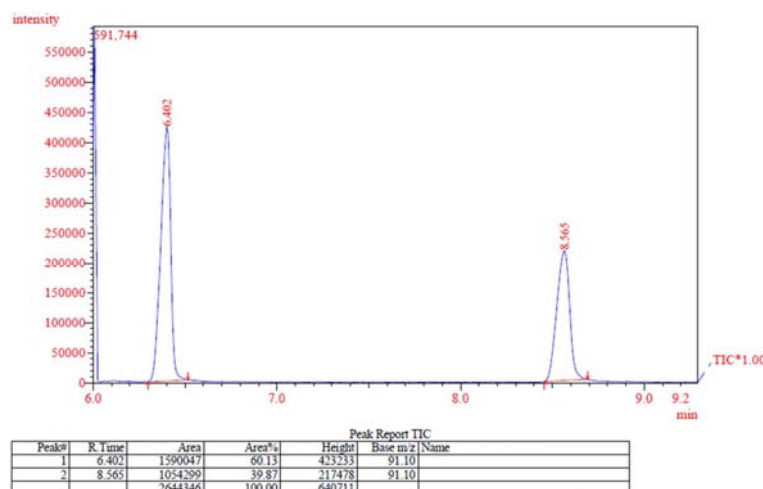


Fig. 7: Chromatogram of recycled polymer

3.2. Chromatographic Differentiation of vPET and rPET

Comparative chromatographic analysis demonstrated distinct structural and chemical differences between virgin and reprocessed polymer matrices.

- **Virgin Polyester Profile:** As shown in the representative chromatogram, vPET displays a single, sharp analytical response at retention time (Rt)=6.386 min corresponding to the added standard reagent, with complete absence of secondary volatile degradation products or unreacted monomeric peaks in the extended retention region.
- **Recycled Polyester Profile:** In contrast, the chromatogram for the reprocessed material shows the primary standard peak at retention time (Rt)= 6.402 min alongside a distinct secondary peak at Rt=8.565 min (Area %=39.87%). This secondary peak corresponds to liberated residual IPA. The appearance of this characteristic secondary peak (Rt≈8.56 min) serves as an analytical indicator of reprocessed PET content. During mechanical recycling, repeated thermal cycles and moisture-induced hydrolysis promote chain cleavage, releasing esterified IPA co-monomer units into free or low-molecular-weight oligomeric forms that are readily extracted and identified during derivatization.

3.3. Toxicological and Regulatory Considerations

It is critical to distinguish between total internal residual content (wt%) and specific chemical migration limits (SML) into contacting media. Regulatory frameworks (e.g., EU Commission Regulation No 10/2011) mandate that compliance for food-contact plastics be determined by mass transfer into liquid simulants rather than total constituent concentrations within the polymer matrix (7, 8). While internal concentrations of IPA average 2.00 wt%, actual health hazards depend on migration rates under intended conditions of use. Consequently, quantitative identification of IPA serves primarily as a quality and structural marker rather than direct evidence of toxicological non-compliance.

4. Conclusions

Gas chromatographic characterization following depolymerization and derivatization provides a reliable analytical method for evaluating co-monomer concentrations and processing history in polyethylene terephthalate (PET) materials. The quantitative analysis established a baseline average isophthalic acid (IPA) content of 2.00 wt% across common commercial food-contact packaging. Furthermore, comparative chromatographic profiling demonstrated clear differentiation between virgin and recycled matrices based on the presence of a characteristic residual IPA peak at retention time (R_t) = 8.565 min in reprocessed samples. This analytical protocol

provides a useful approach for material verification, quality control, and supply chain audit within circular polymer manufacturing.

Conflict of Interest

The authors declare no competing financial or personal interests.

Acknowledgments

The authors express gratitude to The Bombay Textile Research Association (BTRA) for providing access to the instrumentation and analytical facilities necessary for this investigation.

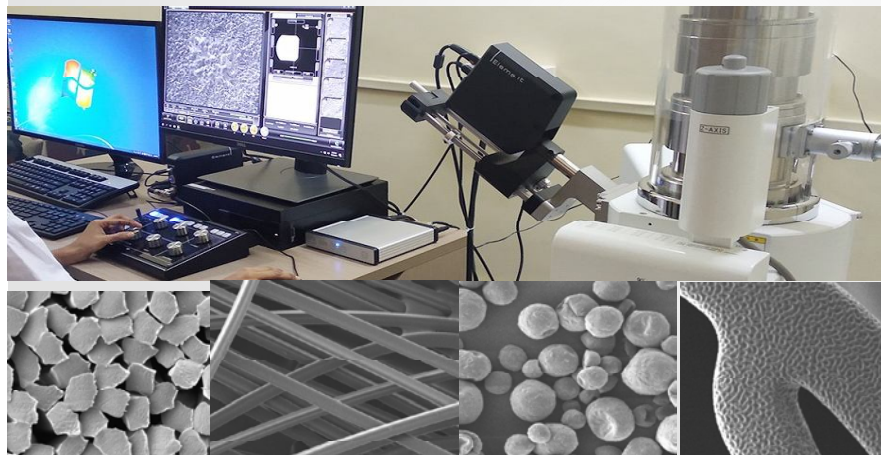
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Developments of standard procedure to identify dope dyed and exhaust dyed meta aramid yarn

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Abstract

Indian Air force uses meta-aramid overall for their pilots due to its excellent properties such as high strength, flame and chemical resistance, etc. Dyeing of those overall materials is recommended through a dope dyeing process to achieve good fastness properties and uniform color. Dope dyeing is the process of adding color during spinning, while in the exhaust dyeing process, dye is gradually transferred from dye bath to textile materials. However, the fabric of the same color with different preparation routes looks similar but behaves differently during actual application due to different fastness properties. No standard method distinguishes exhaust or dope-dyed meta-aramid fiber/fabric. So, in this work, meta-aramid samples were dyed using both the dyeing process and different properties were investigated to find a confirmative method to distinguish the dyeing process. Based on the investigation, the heat treatment method and light fastness is found to be most suitable for this. The overall of Air Force pilot was also tested by this method for confirmation.

Key words:

Exhaust dyeing, Dope dyeing, Meta-aramid, Dyes and Pigments

Citation

Komal Kukreja, Prasanta K. Panda*, "Developments of standard procedure to identify dope dyed and exhaust dyed meta aramid yarn", *BTRA Scan* - Vol. LV No.4, Oct., 2026, Page no. 10-14, DOI: 10.70225/822591cvdzy

1. Introduction:

Different types of colored textile materials are available in the market. These textile materials are based on natural or synthetic materials. Synthetic materials are gaining wide acceptance due to their low cost and better properties. The most widely used synthetic fiber for flame retardant textiles is meta-aramid (MA) because of its excellent properties, such as high strength, abrasion resistance, and high-temperature resistance [1]. They are colored through either dope dyeing during the spinning stage or exhaust dyeing after producing filaments/fibers for specific applications [2].

Dope dyeing of filaments/fibers has gained considerable interest in the defence and industrial community in recent years [3]. It is primarily due to the intrinsic dyeing of fiber during spinning. It has altogether better physical and chemical properties than exhaust-dyed material [4]. Dope-dyed meta-aramid yarn is preferred in specific application areas like Army uniforms in Defence due to its higher fastness properties [5],[6]. In this context, it has also been realized that doped dyed polymer puts less burden on the environment. Despite this, the textile processors might compromise on this front because of the marginal increase in profit. Therefore, exhaust dyeing is still prevalent in the

textile wet processing industry not only due to its lower cost and easy processability but also because of having many more choices of different colors, unlike dope dyeing, where only fiber/filaments manufacturer have their options with the limited number of color pigments [7].

The meta-aramid fabric of the same color but produced by dope [8] and exhaust-dyeing routes [9] looks similar. However, the performance of the dope-dyed and exhaust-dyed fabric in terms of color fastness to various chemical agents and environmental conditions is different [10], [6]. Consequently, this difference in fabric performance may create shade-off or a tonal variation, which creates hurdles to achieving the desired uniformity [11]. However, no standard method is available to distinguish exhaust or dope dyeing methods of meta-aramid fibers/fabrics. Efforts are being made in this direction, but no confirmative result has been reported yet [12], [13]. Therefore, it is necessary to develop a standard method to understand whether the sample is dope dyed or dyed using the exhaust dyeing route.

In this research work, we have prepared colored samples through dope dyeing and exhaust dyeing processes. Systematic investigations of dope-dyed and exhausted yarns have been conducted to find a confirmative method to differentiate the dyeing process.

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2. Materials and Methods

2.1 Materials

Meta-aramid dope dyed and raw white yarn was supplied by Arvind India Ltd., India. Coracryl Red Modified Basic dye was purchased from Colourtex Industries Pvt. Ltd., India. Sodium carbonate and acetic acid were procured from Merck, India. Dimethyl formamide (DMF) and sodium dithionite were procured from Loba Chemie Pvt. Ltd., India. All the received chemicals were used as they received.

2.2 Methods

2.2.1 Exhaust Dyeing of raw white yarn

Aramid yarns were dyed with cationic dye. The pretreatment process was carried out before the dyeing so that the complex structure could open up and facilitate dyeing [14], [15]. In this process, aramid yarn was pre-treated with DMF with MLR 1:15 at a temperature of 80°C for 15 minutes and then washed with hot water. It was dyed with Coracryl Red Modified Basic dye with 5% shade, MLR 1:35, and pH 4-5. Dyeing was done in a high-temperature, high-pressure beaker dyeing machine at 120°C for 1 h followed by treatment with 1 gpl sodium carbonate and 1 gpl sodium dithionite at 100°C for 15 minutes and subsequent regular wash [9].

2.2.2 Spectral Colour Analysis

Spectral colour analysis was done before and after testing to observe any change in colour of the sample. Macbeth Colour Eye 7000 A Spectrophotometer were used to analyze the color value of the exhaust-dyed and dope-dyed yarn. Testing was done in UVD65 light under the specular included condition.

2.2.3 Dye Stripping

Dye stripping tests were conducted on exhaust-dyed and dope-dyed aramid yarn specimens to assess the retention and removal of the colorant. The stripping procedure was performed in a solvent DMF at temperature 80°C for a period of 30 minutes. Following the treatment, the samples were removed, rinsed thoroughly with distilled water to remove residual solvent and detached dye, and then dried at room temperature until a constant weight was achieved. Visual observation and colour value measurement were done to observe change in colour.

2.2.4 Colour Fastness to Sublimation

Meta-aramid fabric samples from both exhaust-dyed and dope-dyed materials were cut and placed in contact with a standard multifiber adjacent fabric DW. The dyed specimen and multifiber was sewn or secured together to maintain intimate contact. The prepared samples were subjected to a sublimation fastness test by placing them between heated plates for 30 seconds at temperature 180 and 210°C in Preso Hot machine. After heating, the samples were allowed to cool. Samples were then analyzed for staining in adjacent

multifiber fabric, and the specimen's color changed with time.

2.2.5 Colour Fastness to Washing

Specimens of the dyed meta-aramid yarn were prepared for testing color fastness to washing. Each sample was cut and mounted onto a multifiber adjacent fabric. The color fastness to washing was determined using the standard ISO 105 C10:2006 (Test Method B, C, E), which simulates a severe wash condition typically seen in industrial or protective garment cleaning. Test conditions for different method is listed in Table 1. The test was conducted in a laboratory washing machine Roaches Washtec-P. Following the wash, the composite samples were rinsed thoroughly under cold running water, hydro-extracted, and dried at room temperature. Samples were then investigated for staining in adjacent fabric and change in sample color.

Table 1 : Parameters for Washing Fastness Test

Method	B	C	E
Temperature (°C)	50	60	95
Time (min)	45	30	240
Steel balls (No.)	0	0	10

2.2.6 Colour Fastness to Light

The color fastness of the dyed meta-aramid samples was assessed by exposure to a Xenon arc light source in Q-Sun Xe-3-HDS Xenon Test Chamber . Specimens of the dyed yarn were mounted onto cards. The test was conducted by progressively exposing and covering sections of the test specimen for a period of 4 hours. These were then analyzed for fading of the sample.

2.2.7 Heat Treatment

Samples dyed with different methods were heated to the temperature of 300°C for 10 minutes in muffle furnace and then compared with the color value of the original dyed sample for any change in color or tone.

3. Results and Discussions

3.1 Dye stripping from dyed samples

Dye stripping is one of the simplest and most reliable tests to distinguish exhaust-dyed yarn from dope-dyed yarn because the way color is incorporated into the fiber is fundamentally different. The dye stripping from both dope-dyed and exhaust-dyed samples of meta-aramid was carried out in DMF. The treated samples are shown in Figure 1 and the reduction in color strength value for both samples after stripping was analyzed as shown in Table 2. The dye bleeding from both the sample takes place, but it is observed minimal in dope-dyed samples but significant in exhaust-dyed sample. The significant color change demonstrate that the dye in exhaust-dyed aramid is more accessible to the solvent. Meta-aramid fibers are known to have a highly thermostable crystalline structure with strong intermolecular forces.

While polar solvents like dimethylformamide (DMF) can swell the structure by forming hydrogen bonds with the aramid chains [16]. This can facilitate dye removal from the surface and amorphous regions. However, In dope dyeing, the colorant is added to the spinning solution (dope) prior to fiber formation, thus dye is mechanically encapsulated and locked within the crystalline molecular matrix of the aramid fiber as it solidifies during the spinning process. This internal encapsulation shields the dye from chemical attack, making it highly inaccessible to the stripping solvent (DMF) under the test conditions. Therefore, the stripping test acts as a chemical diagnostic tool to differentiate between the surface-fixed nature of exhaust dyeing and the matrix-embedded nature of dope dyeing, but the amount of dye stripping may mislead to confirm if stripping is from dope dyed and exhaust dyed samples. Thus it can not be confirmative test methods, however it can be a supporting test method with other test methods.



Figure 1 (a) Exhaust dyed sample, (b) Dope dyed sample before (left) and after (right) dye stripping test

Table 2 : Color Value (K/S) of samples before and after stripping

Sample/ Colour Strength	Before Stripping	After Stripping	Change in Colour Strength%
Dope Dyed Yarn	6.52	5.93	9.1
Exhaust Dyed Yarn	10.41	6.41	38.4

3.2. Analysis of dye sublimation

A sublimation fastness test evaluates the stability of the color in a dyed testiles when exposed to high temperature and dry heat. It checks whether the dye vaporizes, migrates, or transfers to an adjacent fabric. Color fastness to sublimation was performed on dope dyed and exhaust dyed samples at temperature 180° and 210° C for 30 seconds. The tested samples is shown in Figure 2. It is clear fom the figure that no staining in adjacent fabric is observed. This is because aramid fiber is resistant to heat, due to which it does not get affected with testing temperature.

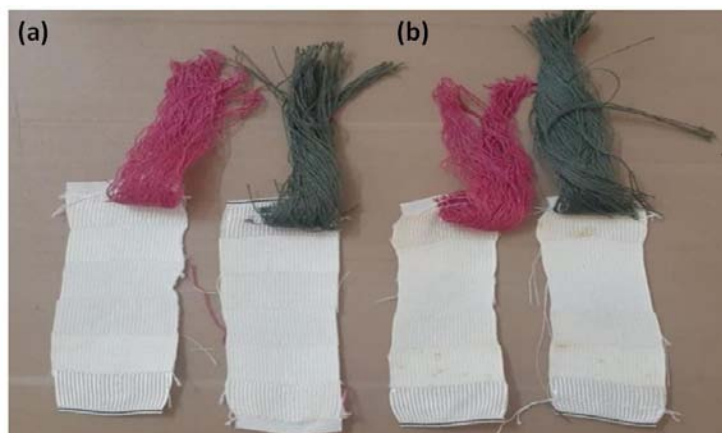


Figure 2 : Exhaust dyed (left) and dope dyed (right) sample at (a) 180°, (b) 210°C of sublimation fastness test

3.3. Analysis of color fastness to washing

Color fastness to washing was carried out to differentiate dope-dyed and exhaust-dyed samples because dope-dyeing incorporates color within the fiber core, resulting in superior retention during washing. In contrast, exhaust-dyed materials may have surface adsorbed or partially fixed dye that can wash off, leading to shade change and staining. The color fastness to washing of meta-aramid samples were investigated by using IS/ISO 105 C10:2006method. The samples tested with method B and C is shown in Figure 3. It is clearly seen from the figure that no staining in adjacent fabric was observed. Again this is due to the fact that mild conditions of washing does not have any impact on aramid yarn.



Figure 3 Exhaust dyed (left) and dope dyed (right) sample tested with (a) Method B, (b) Method C

3.4. Analysis of color fastness to light

Colour fastness to light was performed to differentiate between dope-dyed and exhaust-dyed samples because in exhaust dyed samples, dye present on the surface are more exposed to external environmental conditions as compared to the dye present in dope dyed samples, leading to distinct

differences in light durability. The samples were exposed to UV light for 4 hours. However the change in colour starts observing within one hour of exposure as shown in Figure 4(a). The change in the color value of aramid samples concerning exposure time is shown in Figure 4(b). In the case of exhaust-dyed samples, a reduction in color value was observed from 8.4 to 3.7 after 4 hours of exposure while in the case of dope-dyed yarn, no major change in color value was observed. This is because in case of exhaust dyed samples, surface dye interact with the photon of xenon arc light causes photochemical reaction that lead to the degradation of the dye molecule [17].

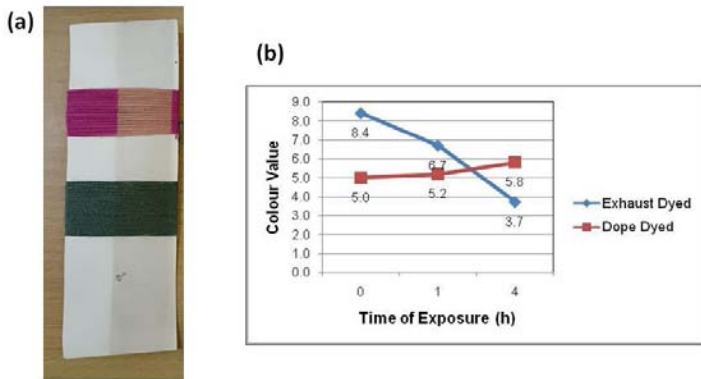


Figure 4 (a) : Exposed sample (b) Colour value (K/S) of exhaust dyed and dope dyed aramid yarn for 4 hours exposure

3.5. Effect of high temperature for a short time

Direct exposure of dye to high temperature leads to the degradation of dye present on the exhaust dyed sample. While colorant or pigments used in dope dyed sample are stable at high high temperature of polymer melting and spinning during its production process. So direct exposure of exhaust dyed and dope dyed samples were done at high temperature of 300°C in muffle furnace for 2 minutes was done to see the effect of high temperature on the colour change was observed. The treated sample of exhaust dyed and dope dyed is shown in Figure 5(a) and (b) respectively and colour value is shown in Table 3. There is clear colour change observed for exhaust dyed sample with decrease in colour value while no such change is observed for dope dyed samples.

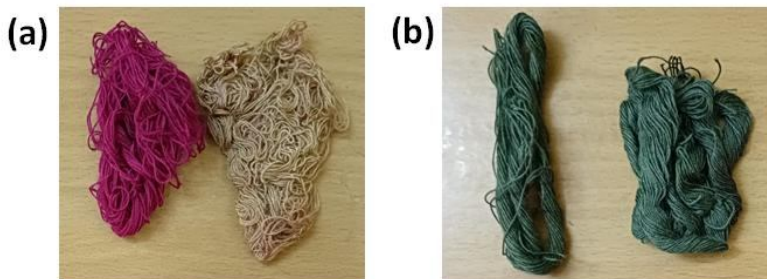


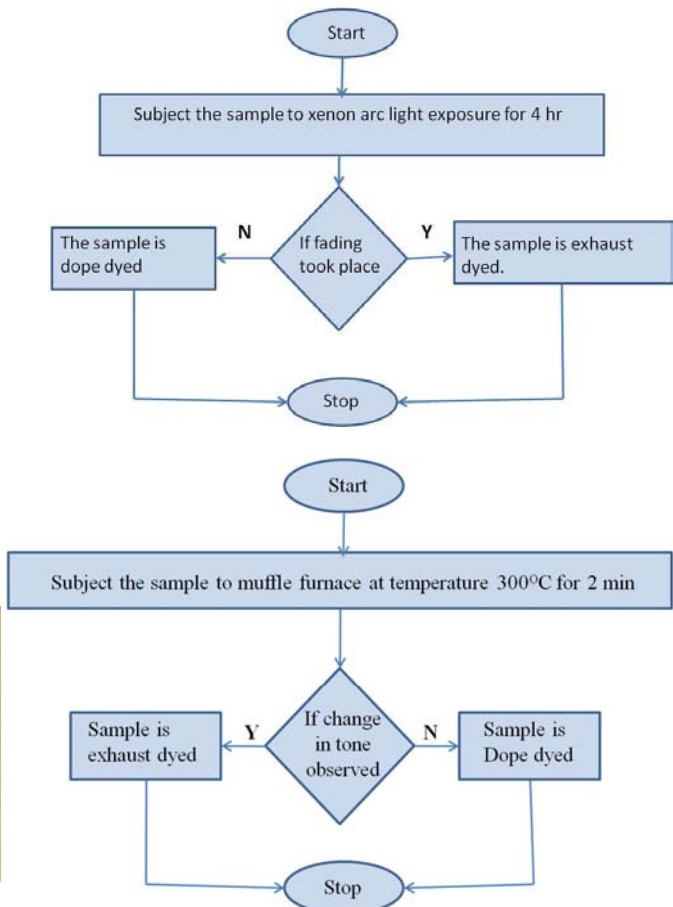
Table 3 : Colour Value of dyed meta-aramid yarn before and after heat exposure

Particulars	Before heat exposure	After heat exposure
Meta-aramid Cationic Exhaust Dyed Yarn	20.73	3.90 (Tone change)
Meta-aramid Dope Dyed Yarn	6.58	6.59

4. Conclusions

The investigations and obtained results conclude that the color change to sublimation and vigorous washing cycle does not provide any conclusive result for identification of dope dyed and exhaust dyed samples. In the other way, color fastness to sublimation and exposure to high temperature for a short period were the turning point of this work to confirm the dyeing process of meta-aramid sample quickly. The complete change in tone of the dyed sample was observed at high temperatures, whereas no change was observed in the dope-dyed sample. Based on this result, a flow chart is shown below for a confirmative quick method to differentiate the dyeing process from the dyed meta-aramid sample within a short time.

Flowchart 1 : Light fastness test



Flowchart 2 : Heating sample in a muffle furnace at temperature 300°C for 2 minutes

Conflict of interest

The authors declare no conflict of interests

Author contributions

KK performed the experiments, including sample preparation and testing. PKP conceptualized the study and supervised the research. All authors contributed to the preparation and review of the manuscript.

Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Transitioning to Green Textile Manufacturing: The Synergistic Role of Microbial Enzymes and Biosurfactants

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Abstract

The textile industry is under severe pressure to replace highly polluting chemical processes with eco-friendly alternatives. While individual applications of microbial enzymes (e.g., cellulases, amylases, pectinases, laccases) and microbially derived biosurfactants (e.g., rhamnolipids, sophorolipids, surfactin) have advanced sustainable textile processing, their combined, synergistic application represents an unmapped frontier. This review comprehensively analyzes the multi-faceted mechanisms driving the synergistic relationship between microbial enzymes and biosurfactants across wet processing operations including desizing, scouring, bleaching, biopolishing, and wastewater remediation. Study explores how biosurfactants enhance enzyme performance by reducing interfacial tension, improving fiber wetting, and stabilizing enzymatic tertiary structures against harsh process dynamics. Simultaneously, enzymatic modification of fabric surfaces exposes localized domains, allowing optimal biosurfactant micellization and localized activity. Finally, we address current techno-economic bottlenecks, scale-up barriers, and future multi-omics-driven strategies required to commercialize this binary biomolecular framework for next-generation circular textile manufacturing.

Key words:

Microbial Enzymes; Biosurfactants; Wet Processing; Interfacial Synergy; Bio-scouring; Textile Wastewater Remediation

Citation

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1.0 Introduction

The modern textile industry is facing significant challenges due to its heavy dependence on resource-intensive wet processing steps such as desizing, scouring, bleaching, and finishing.

These traditional processes use large amounts of fresh water, energy, and synthetic chemicals. Standard production methods rely on strong oxidizing agents, harsh alkalis, and non-biodegradable surfactants, which result in highly alkaline, colored, and ecotoxic wastewater [1]. As global environmental regulations become more stringent and the industry moves towards zero-liquid discharge (ZLD) systems, there is an urgent need to incorporate green chemistry practices into the core operations of the textile sector [2].

Industrial biotechnology presents a promising solution to this environmental shift.

Specific microbial enzymes function as targeted biocatalysts that can safely replace harmful synthetic additives. At the same time, biosurfactants produced by microorganism's surface-active amphiphilic secondary metabolites grouped into lipopeptides, glycolipids, and polymeric complexes have become a sustainable alternative to surfactants derived from fossil fuels. These bio-based alternatives have excellent biodegradability, low ecological toxicity, and stability under a wide range of temperature and pH conditions [3].

Importantly, using these biological components on their own often causes operational issues in industry.

When used alone, enzymes may require long processing times, high dosages, or fail to fully modify the fabric. Recent developments show that combining microbial enzymes with biosurfactants results in a powerful, multi-step physical and chemical synergy that overcomes the limitations of using them individually, opening up a new area in environmentally friendly textile processing [4].

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2.0 Molecular Profiles of the Biomolecular Partners:

2.1 Textile Microbial Enzymes

Microbial enzymes act as precise tools, classified based on their ability to identify and break specific chemical bonds in both natural and synthetic fibers:

Amylases (α -amylases): These biocatalysts quickly break down the internal α -1,4-glucosidic bonds in starch-based sizing agents, transforming insoluble polymer networks into water-soluble sugars that can be easily rinsed away [5].

Pectinases: These enzymes break down the complex pectin matrix within the primary cell wall of raw cotton fibers.

By breaking this structural component, they uncover trapped natural waxes and non-cellulosic impurities for further removal.

Cellulases (Endoglucanases): These agents specifically break the internal β -1,4-glucosidic bonds in the amorphous parts of cellulose.

This controlled breakdown is essential for removing surface fuzz (biopolishing) and achieving the popular look of distressed denim through stonewashing [8].

Laccases and Peroxidases: These multi-copper enzymes use radical-based oxidation pathways to directly oxidize natural pigments during bio-bleaching and break down complex, stable azo-dye chromophores during wastewater treatment.

2.2 Microbially Derived Biosurfactants

Biosurfactants are primarily produced during the stationary phase of microbial fermentation.

They have a unique amphiphilic structure with a hydrophilic (polar) head and a hydrophobic (non-polar) hydrocarbon tail [4].

Glycolipids: This group includes rhamnolipids made by *Pseudomonas aeruginosa* and sophorolipids produced by *Starmerella bombicola*.

They are known for very low critical micelle concentrations (CMCs) and strong emulsifying abilities, making them effective mobilizing agents [6].

Lipopeptides: This group includes surfactin and iturin, made by *Bacillus subtilis* through non-ribosomal peptide synthetases [7].

They have a strong ability to reduce water surface tension and lower the interfacial tension between immiscible substances [8].

3.0 Mechanisms of Interfacial & Catalytic Synergy:

Without Biosurfactants	With Biosurfactants (Sub-CMC)
High Surface Tension	Drastic Surface Tension Drop
Enzymes blocked by wax barrier	Rapid fabric wetting & fiber swelling
Slow, uneven catalytic attack	Enzyme structure stabilized & protected
Reprecipitation of hydrophobic debris	Complete micellar encapsulation of debris

The synergistic interaction between enzymes and biosurfactants results from a mutual physicochemical process occurring at the interface between fiber and water [9].

Natural fibers like unprocessed greige cotton have a natural hydrophobic coating made of resins, fats, and waxes. This organic layer acts as a physical barrier, preventing aqueous enzyme solutions from reaching their intended targets.

To counter this, biosurfactants quickly arrange themselves at liquid-gas and liquid-solid interfaces, reducing water surface tension from about 72 mN/m to below 30 mN/m [8, 9].

This significant decrease allows the processing liquid to quickly wet and penetrate the fabric, improving the physical movement and attachment of large enzyme molecules onto the fiber surface [9].

When added at concentrations below their critical micelle concentration (sub-CMC), monomeric biosurfactants act as structural helpers for the enzymes.

The hydrophobic parts of these single monomers connect gently with the non-polar areas on the enzyme's surface, while their hydrophilic parts face outward into the water [10].

This specific organization helps preserve the enzyme's fragile three-dimensional structure, stopping it from unfolding or losing its shape in harsh, hot environments used in textile processing. Also, these specific connections can help the enzyme stay in a flexible and active shape, directly increasing its ability to perform its function [10].

During processes like enzymatic desizing or scouring, leftover material from degradation (such as broken-down waxes, fatty acids, and short-chain dextrans) often sticks back to the clean fabric due to strong hydrophobic forces [11].

Biosurfactants help with this problem. As their amount increases, they form spherical structures called micelles that trap these hydrophobic materials inside their centers, stopping them from returning to the fabric [11].

Textile Process	Target Impurity	Primary Enzyme(s)	Biosurfactant Class	Synergistic Outcome
Desizing	Starch sizes, lubricants	alpha-Amylase	Lipopeptides (Surfactin)	Accelerated size hydration; 30% reduction in processing time.
Scouring	Cuticular waxes, pectins	Pectinase, Lipase	Glycolipids (Rhamnolipids)	Rapid wax emulsification; uniform absorbency at low temperatures.
Biopolishing	Surface micro-fibrils	Endoglucanase	Sophorolipids	Controlled surface shaving; prevents excess strength loss.
Effluent Treatment	Azo & anthraquinone dyes	Laccase, Azoreductase	Polymeric/ Glycolipids	Enhanced dye bioavailability; rapid breakdown of aromatic rings.

4.0. Industrial Applications in Textile Wet Processing:

Using pectinases and amylases together with surfactin or rhamnolipids lets textile mills combine several steps into a single, efficient pre-treatment process.

In this method, the biosurfactants quickly loosen and mix the outer cuticular waxes, giving pectinases direct access to break down the pectin layer beneath.

Similarly, adding glycolipids or sophorolipids during laccase-based bleaching and cellulase-mediated biopolishing reduces the loss of strength in the yarn.

By guiding how the enzyme attaches to the fiber, the biosurfactant prevents it from deeply affecting the strong cellulose core, ensuring it only removes the unwanted surface fuzz [12].

5.0 Biodegradation and Effluent Treatment:

Cleaning up wastewater from textile plants is very hard because of high levels of chemical oxygen demand (COD), strong color, and the presence of harmful aromatic amines from broken-down azo dyes [13].

Hydrophobic dyes tend to stick together in water, forming clumps that block enzymes like laccases from reaching the dye molecules.

Adding biosurfactants breaks these clumps apart and traps individual dye molecules inside micelles.

This increases the dye's surface area, making it easier for enzymes to break it down [13]. Importantly, this method avoids creating toxic chemical sludge that traditional methods leave behind, providing a cleaner and more eco-friendly solution [14].

Key Benefits of Using Biosurfactants:

- Increased Bioavailability: Micelles break up dense dye clusters, making more of the dye available for enzymes [13].

- Faster Reaction Rates: Enzymes can break down isolated dye molecules at a much quicker pace [13].
- No Extra Waste: This process avoids the heavy, harmful sludge that comes from standard chemical treatments [14].

6.0 Current Challenges and Future Outlook:

Even though this approach has great environmental benefits, high production costs and the risk of enzyme damage are holding back its wide use.

Low production levels for biosurfactants make them more expensive than cheaper, petroleum-based alternatives [4]. Also, mixing these systems needs exact control; high doses of ionic biosurfactants can disturb the enzymes' internal structure, causing them to unfold and stop working [10].

To make this technology work for industry, future research must focus on three areas:

- Using Synthetic Biology for Strain Development: Creating advanced microbial factories that can produce and release both enzymes and biosurfactants in the same liquid during fermentation.
- Using Advanced Molecular Modeling: Using detailed computer simulations to study how biosurfactants and enzymes interact, helping predict the best mix without testing all options.
- Using Biomolecular Immobilization: Attaching both enzymes and biosurfactants to strong, reusable solid materials or magnetic nanoparticles to make them last longer and easier to recover from the processing water [15].

7.0 Conclusion:

The collaboration between microbial enzymes and biosurfactants marks a significant advancement in the development of sustainable textile manufacturing.

Together, they enhance fabric wetting, support protein structure stability, speed up catalytic processes, and help break down difficult processing byproducts. This combined system provides a strong alternative to harmful chemical treatments. Adopting this integrated biological approach is essential in guiding the global fashion and textile industries away from traditional, fossil-fuel-based methods and towards a cleaner, circular bio-based economy.

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9.0 Authors and Affiliations:

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11.0 Conflict of Interest:

The author declares no conflict of interest.

12.0 Data availability statement:

Data sharing is not applicable to this article.



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