

Improved Sustainable, Environment Friendly, Green Technology For Textile Dyeing Using Supercritical Fluid

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Abstract

Deven Supercriticals, India (DSPL) has developed innovative Supercritical (SC) Carbon Dioxide (CO₂) based dyeing and finishing technology that is uniquely suitable for not only polyester but also for cotton and blended textiles. Further, it uses conventional dyes (No special dyes required) and recipes as used in the conventional process, to get the desired shade BUT without the use of water in the dyeing process. This innovative process shows improved dye utilization, makes scale-up easy and has less than half dyeing time vis-a-vis prior-art SC CO₂ based dyeing processes. There is no need for reduction clearing for polyester; cotton with no salt added, dyes blends in a single step, reduces overall auxiliary chemicals. Thus substantially reducing the pollution, water and energy load. This has truly made the SC CO₂ technology viable, versatile and simple.

Keywords

Sustainable Dyeing, Water-less Textile Dyeing, Supercritical, Carbon Dioxide, SCF, Green Technology, Wet processing

1. Introduction

Traditionally, water has been a popular medium used in dyeing, finishing, cleaning textile materials. It makes the textile industry one of the largest consumers of water resources. On the other hand, the cost of input water and wastewater treatment is ever increasing along with the pollution control norms becoming more stringent each year. Also globally, usable water resources are becoming alarmingly scarce. Recently in December 2020, water has even started trading on Wall Street as a 'Futures commodity' to join the likes of Gold and Oil.

In this regard, it has become very critical that Textile processes that use minimum or no water are developed and adapted on a commercial scale. In recent years, the use of supercritical fluids as a replacement for water as a solvent, in the Dyeing process has attracted the attention of the Textile industry. Carbon Dioxide (CO₂) has emerged as the most preferred supercritical solvent. Major advantages of Supercritical CO₂ (SC CO₂) based Textile Dyeing process which also improves its 'Economic Viability' and 'Consumer preference' are as follows:

- 1) Zero discharge: Elimination of waste water streams, Pollution.
- 2) Shorter process and dyeing times because:
 - i) SC CO₂ penetrates in the polymer matrix and swells it to help in faster diffusion of dye molecules within the polymer matrix.
 - ii) SC CO₂ has negligible surface tension resulting in efficient wetting of polymer surface and faster penetration in voids of textile material.
 - iii) SC CO₂ has a low viscosity which helps in efficient and easy circulation of the solution of SC CO₂ and dye, through the textile material.
 - iv) SC CO₂ has higher diffusivity which helps in faster mass transfer.
- 3) Efficient process because of Lower dye consumption, no wastage and dye can be reused.
- 4) Energy saving process due to minimum requirement of expensive 'heat energy' and resource required for post dyeing repeated water washing and drying of dyed fibre or fabric.
- 5) SC CO₂ is recyclable. inert, nonexplosive, Generally Regarded as Safe (GRAS) solvent.
- 6) There is no damage to the fibre or fabric.
- 7) Many pre and post treatments of textile material are simplified or eliminated.

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Limitations of Prior Art Technology / Motivation for Innovation:

The conventional/prior art supercritical dyeing processes that were available in the world had the following major limitations which have also resulted in limited adoption of the said prior art supercritical fluid-based sustainable processes:

- (i) The dye needs to be first dissolved in SC CO₂ and then transported to the Textile in placed in a Dyeing vessel.
- (ii) Dyes have Low solubility in SC CO₂ resulting in low dye concentration in the dye solution.
- (iii) The low residence time of dissolved dye flowing through the Dyeing vessel, limits the contact, interaction of Textile material with dye molecules.
- (iv) Some part of dissolved Dye that is flowing through the 'Dyeing vessel' may not come in contact with the Textile surface. Also, Non-uniform flow / Channelling of SC CO₂ solution through a Textile roll in a Dyeing vessel can lead to non-uniform contact and thus non-uniform dyeing in large scale operation. Thus, it may require special, complicated additional devices to impart say rotational motion to the textile roll in dyeing vessel, to improve the uniformity in dyeing.
- (v) Thus, only part of the available dissolved dye may take part in SC CO₂ Dyeing to achieve desired colour Intensity on Textile material.
- (vi) The final shade of dyed cloth depends on the extent of exposure as the shade keeps getting darker with the passage of contact time with fresh dye solution entering the Dyeing vessel, making it difficult to control Batch Batch variation.
- (vii) Mainly useful for applying Dark shades with a single colour at a time.
- (viii) All the above limitations make the prior art SC CO₂ dyeing process Less versatile, Slow and Less efficient.

Hence, the objective of innovative work at DSPL was to develop an improved dyeing process:

- To get Uniform, Reproducible Interaction between Dye molecules and the entire surface of Textile material.
- To Improve the rate of Solubilisation of Dye molecules in Supercritical CO₂ solvent to increase the Rate and Efficiency of the SC CO₂ dyeing process.
- To achieve easy scale-up to large scale dyeing

while maintaining desired Uniform & Reproducible Colour intensity on textile material.

- To get dyeing of the textile materials with a single or multi-colours in various shades, patterns etc. in a single step of dyeing operation.

2. Methods & Materials:

2.1 Details of Innovative SCF Dyeing Process from DSPL:

Innovation has been carried out by following steps:

Making a dye solution: By mixing the dye material and auxiliary chemicals with a suitable solvent. We preferably use water as a solvent for the conventional dyes along with the dispersing, levelling agents.

Pre-treatment: Pre-coating the surface of textile material to be dyed with an optimum quantity of the above dye solution to obtain a dye coated textile material. Any standard method of coating can be used such as Roller coating, Ink-Jet Printing etc.

Supercritical CO₂ process: Placing dye coated textile material inside the supercritical 'Dyeing vessel' on a supercritical fluid processing plant.

Adding the supercritical CO₂ into the 'Dyeing vessel'. Exact operating conditions are optimized as per the type of dye, auxiliary chemicals and textile used, wherein the supercritical CO₂ solubilizes the dye molecules that were earlier coated on the surface of the textile material and further diffuses the solubilized dye molecules inside the surface, pores and capillaries of the textile material;

Depressurizing the supercritical fluid dyeing vessel to precipitate and entrap the dye material inside the textile material.

Post-Treatment: Mild soap washing of the dyed & finished textile with and Stentering.

2.2 Innovative Elements of Patented Process from DSPL:

Novel / Inventive step: Pre-coating of textiles to be dyed, with the optimum quantity of dye & auxiliary chemical molecules, per unit area of textiles to increase the surface area of solute and improve the rate of solubilisation of dye & other molecules in supercritical CO₂. This also improves uniformity, reproducibility of dyed shade, washing fastness and finishing effect.

Non-Obviousness: Use any pre-coating method such as inkjet printing, Roller coating or similar process for having a controlled pre-coating of optimum quantity of dye molecules, auxiliary chemicals on the textile material to achieve a single or multi-color/light or dark shade dyeing of textiles with post-processing with SC CO₂.

Industrial applicability: Patented improved technology from

DSPL eliminates major limitations of prior art Supercritical dyeing processes available in the market. Its innovative features make it very simple, easily scalable, most efficient and economically viable, to truly achieve the sustainability goals of the user industry.

3. Results and Discussion:



Fig. 1 Photos of 'R-Elan GreenGold' # polyester fabric dyed with disperse dyes with SC CO₂ based patented process from DSPL

(# 'R-Elan GreenGold' is a brand of Reliance Industries, India, for a special Polyethylene Terephthalate (PET) fabric from recycled PET bottles to address environmental pollution)

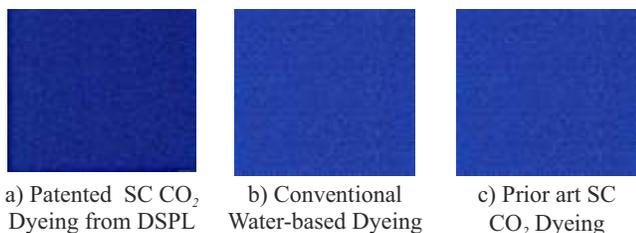


Fig. 2 Photos of R-Elan GreenGold polyester fabric dyed with Navy Blue (1.3 % Shade) using Coralene Navy Blue 3G H/C disperse dye from ColourTex using three different dyeing processes

As is seen in Figure 2, Patented SC CO₂ Process from DSPL gives 18 to 24 % darker colour shade on same "GreenGold" fabric, as compared to samples obtained from conventional water-based dyeing as well as Prior art supercritical dyeing (with same dye & same quantity of dye being used).

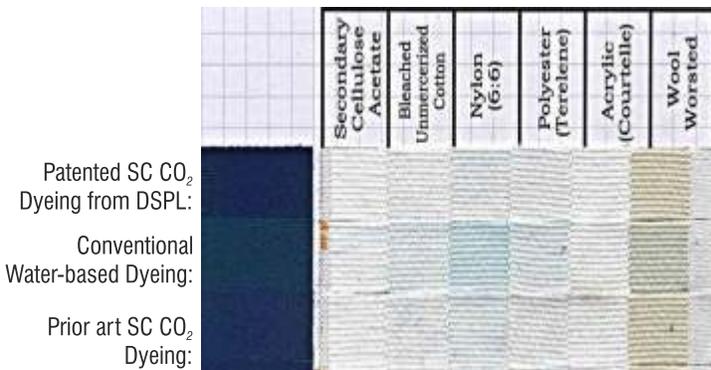


Fig. 3 COLOUR FASTNESS Results as per ISO:105:E01, for dyed GreenGold Fabrics as stated in Fig. 2

As seen in Figure-3 innovative, patented process from DSPL is more efficient and gives better Colour, Washing Fastness.



Fig. 4 Photos of SORONA# Polyester fabric dyed using disperse dye Dianix Navy XF2 from DyStar using two different dyeing processes

"Sorona" is DuPont's brand for an eco-efficient performance Polyester produced by using one of the monomers: 1,3-propanediol, which is obtained from renewable (Plant-based) sources.

As seen in Figure-4, the improved SC CO₂ Dyeing and Finishing Process from DSPL matches the required Navy Blue shade with about 30 % less Dye as compared to the conventional Water-based dyeing process, carried on the same Sorona fabric.

Dyeing of Micro-Denier Polyester Fabric with Patented Process from DSPL:

The micro-denier polyester fabrics have a very high surface area, which poses challenges in dyeing with the conventional Water-based dyeing process. It shows problems like unlevelled dyeing, lower colour depths, lower washing fastness etc. with regular types of dispersed dyes. The above issues are sorted by improved, patented SC CO₂ Dyeing technology from DSPL. As seen in the right side photo of micro-denier polyester fabric (Microsupersoft (125/288), Plain Interlock) dyed with our technology using regular disperse dye (0.75 % Shade of Golden Yellow GG 200%, from Spectrum). A very uniform, levelled dyeing achieved with good colour depth and excellent colour fastness to washing of 4-5.





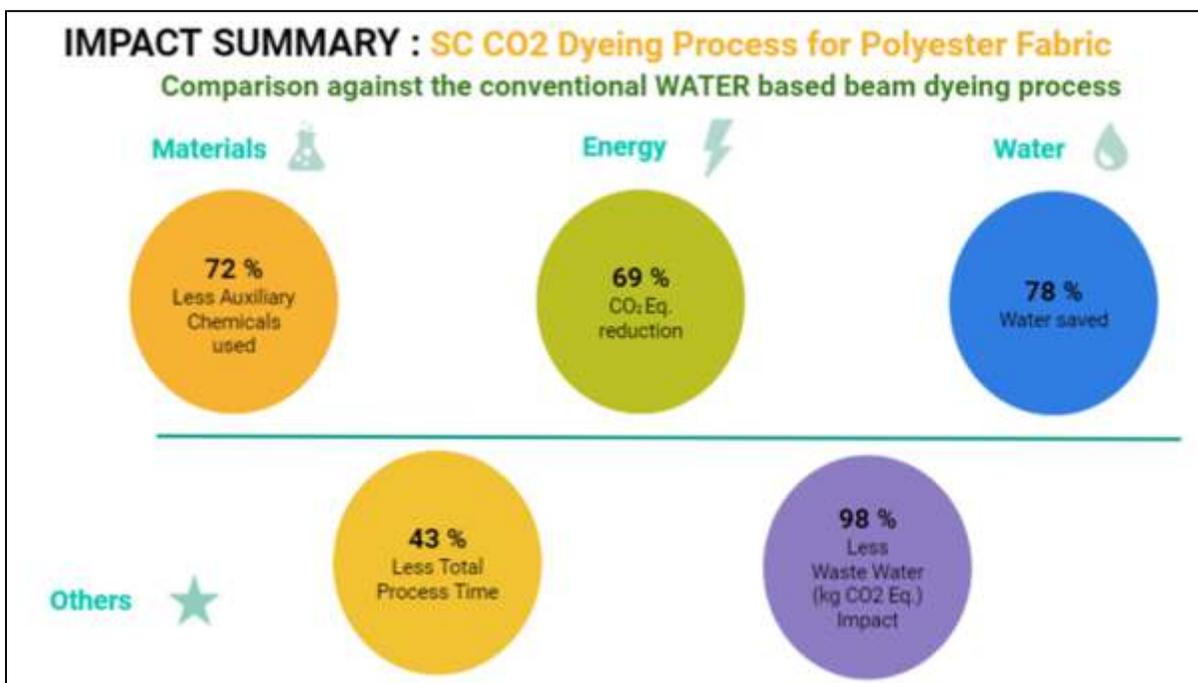
Fig. 5 Photos of Cotton and Polyester-Cotton Blend Dyed with SC CO2 based patented process from DSPL

Results of Life Cycle Analysis (LCA) carried out using 'GaBi' software :

INNOVATION	FEEDSTOCK/INPUT	END OF USE	CERTIFICATIONS
Patented, Most Efficient Supercritical (SC) CO ₂ based sustainable single step Dyeing as well as Finishing technology	<ul style="list-style-type: none"> Innovative SC CO₂ based dyeing from DSPL is suitable for man-made, natural and blended textiles. Allows use of traditional dyes with Improved dye utilisation Finishing chemicals can be applied along with dyeing in single step Much lesser quantity of Auxiliary chemicals required as compared to conventional water based dyeing 	The End of Use would depend on the type of fabric used for dyeing	None at this stage. We have 3 rd party Test Reports from DuPont, ColourTex, BTRA, about the efficient dyeing and Colour Fastness by innovative technology from DSPL

'SCREENING LCA' OUTCOME: SC CO₂ Dyeing Process for Polyester Fabric
 Comparison against a conventional WATER based beam dyeing process

Name	Beam Dyeing with WATER (kg CO ₂ eq.)	SC CO ₂ Dyeing (kg CO ₂ eq.)	% Impact Reduction
Process Water	0.0640	0.0140	78
Electricity	0.0150	0.0090	40
Steam	1.0000	0.3100	69
Acetic Acid	0.0130	0.0005	96
Disperse Dye	0.0850	0.0680	20
Soaping Agent	0.0092	0.0046	50
Carbon Dioxide	0.0000	0.0470	-
Waste Water	0.4570	0.0110	98
Total	1.6432	0.4641	72



Improved Economic Viability due to Innovation:

Innovative Supercritical CO₂ based dyeing process from DSPL has improved economic viability due to the following important factors:

- 1) Process from DSPL is simpler, versatile & efficient with less than half dyeing time vis a vis Prior-art processes, increasing processing capacity & reducing processing cost.
- 2) We can use conventional dyes traditionally used by industry. Thus not necessary to use expensive special dyes required by prior-art processes, improving viability.
- 3) Here thin, a controlled layer of dye is Pre-coated on the surface of textile to be dyed. This increases the effective surface area of solute (Dye) and thus increases interaction and rate of solubilisation insolvent (SC CO₂).
- 4) With the availability of the optimum and uniform quantity of dye molecules on the entire surface of textile material (in form of pre-coating of the very thin layer), the supercritical fluid efficiently dissolves the dye molecules and make them penetrate inside the textile matrix to achieve uniform and efficient dyeing all over.
- 5) Thus, in the process from DSPL Dye molecules are not required to be transported as a Dye solution in supercritical medium, from the 'Dye-Mixing vessel' to the textile material kept in 'Dyeing Vessel'. Also, contrary to the prior-art process, it does not remain critical for the said dye solution to flow and distribute uniformly, over each part of the role of textile material for achieving uniform, reproducible dyeing, even for lighter shades.
- 6) Pre-coating of the Textile surface with the optimum quantity of dye also minimises wastage of dye in overall dyeing operation. Thus lower dye quantity is required for achieving a specific shade as compared to the conventional dyeing process.
- 7) This also enables efficient Dyeing with desired Uniform, Reproducible colour shade, on man-made, natural or blended textile materials (fibres & fabrics), in a single step.
- 8) It also saves expensive 'heat energy' resources otherwise required for post dyeing repeated water washing and drying of dyed Textiles.
- 9) This is a 'Zero Discharge' process, minimises ETP costs. SC CO₂ solvent is recycled.
- 10) Any dye recovered in 'Separator' can be reused as there is No hydrolysis or degradation of dye in SC CO₂.
- 11) This innovation also makes the scale-up of the Dyeing process easier as desired Dye molecules are already made available on the entire surface of Textile material kept in the Dyeing vessel, minimising the fluid and mass transfer related issues.
- 12) Better premium and higher preference from customers for Genuine 'Eco Friendly', 'Green' dyeing processes: giving major economic and marketing advantage.
- 13) Innovation from DSPL allows dyeing & finishing process with softeners, antimicrobials etc. in a single step. Thus Saves on process steps, chemicals, water, time & energy.

4. Conclusions

Supercritical fluid-based dyeing and finishing technology from Deven Supercriticals Pvt. Ltd., India is uniquely suitable for not only man-made fabrics like polyester, Nylon but also for cotton and blended textiles. Also, it allows the use of conventional dyes with no requirement for special expensive dyes. The same recipe of dyes as used in the conventional water-based process can be used in this innovative process but without the use of water in the dyeing process. It further shows improved dye utilization, makes scale-up easy and has less than half dyeing time vis-a-vis prior-art SC CO₂ based dyeing processes. There is no need for reduction clearing for polyester, no salt added for cotton

dyeing, single-step dyeing possible for blend textiles, reducing the overall requirement for auxiliary chemicals. Thus substantially reduces the pollution, water and energy load. It has made the SC CO₂ technology truly viable, versatile and simple. Thus at present and in the future, the improved, efficient and patented supercritical fluid dyeing and finishing technology from DSPL, which takes care of the limitations of the prior-art SC CO₂ based technologies, has great potential to truly Accomplish Environment friendly, Green objectives of Textile Industries around the world for utilising Sustainable processes vis-a-vis the traditional processes which have a negative impact on health and environment.

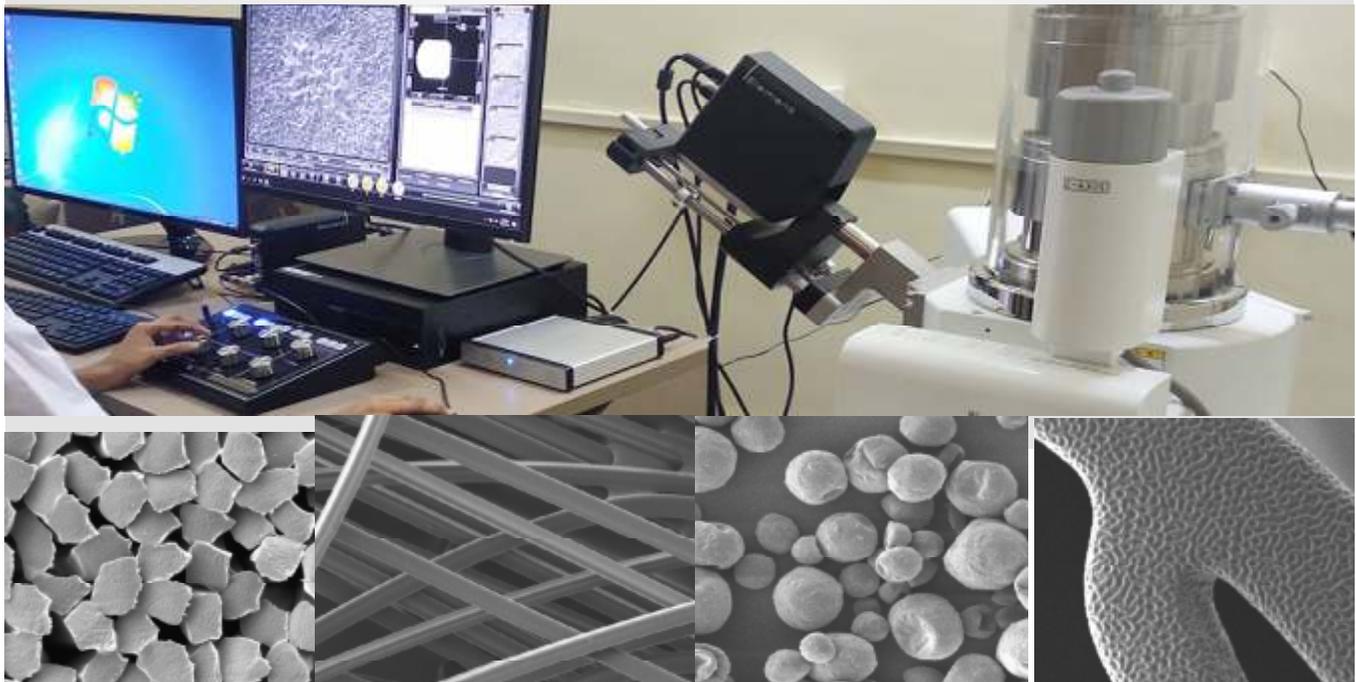
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- 1] "Process for dyeing of textile materials using supercritical fluid", Inventor: Dr Swapneshu Baser, Indian Patent no. 298213 granted in 2018.
- 2] "Process for dyeing of textile materials using supercritical fluid", Inventor: Dr Swapneshu Baser, United States of America Patent No. US 11015289 B2 granted in 2021.

Advanced New JEOL JSM IT 200 LV Scanning Electron Microscope

In BTRA, advanced new JEOL JSM IT 200 LV SEM machine (Japan) have magnification capabilities ranges from 10X to 3,00,000X and resolution of about 10 nm. The surface view and cross-sectional view of the sample can be easily seen. In addition, the elemental composition and mapping of any solid material can be carried out by EDAX (U.S.A.) energy dispersive X-ray spectroscopy (EDS).

Samples from **Textile, Pharmaceuticals, Ceramics, Polymers, Metals and other allied industries** can be analysed on this SEM machine.



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