Modification of polypropylene fibres with
cationic polypropylene dispersion for
improved dyeability

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Polypropylene (PP) fibres are important hydrophobic fibres which are used in the production of functional
textiles such as sports textiles. The absence of functional groups and low polarity make PP fibres difficult to
dye, thus mass coloration during fibre extrusion is the major technique applied today. However, the
disadvantage of mass coloration is the low flexibility and the demand to produce high volumes. A new
method to modify the surface of PP fibres utilises the deposition and thermal fixation of cationic PP
dispersion. Through padding and thermal fixation of a cationic PP dispersion, dyeable 100% PP fibres can be
obtained. The effects of fixation temperature, and of the amount of dispersion used on the modified fibres
were studied using Fourier Transform-infrared spectroscopy, laser scanning microscopy, dyeing experiments
with CI Acid Red 151, and by determining selected fastness properties. The results indicate the potential of
this new method to produce surface-modified 100% PP fibres, which can be dyed in conventional acid-dyeing
processes and therefore used in fibre blends, for example in combination with wool.

Introduction

With annual production at approximately 10 million metric
tons, polypropylene (PP) fibres represent 10% of the total
amount of textile fibres produced alongside polyester,
cotton and polyamide. Because of their hydrophobic nature
and low water sorption, PP fibres occupy a special niche for
applications which require water and sweat transport,
controlled wicking and rapid drying. PP fibre is a polymeric
hydrocarbon-based fibre, thus no polar functional groups
are present in the molecules, which reduces the number of
possible techniques for coloration considerably. The major
process for dyeing PP fibres utilises the principle of mass
coloration, in which concentrated preparations of dyes,
mainly pigment dyes, are added to the PP powder and
incorporated into the fibre during the fibre extrusion
process. This permits production of a wide range of
coloured fibres which have high levels of fastness. How-
ever, there are two major disadvantages associated with this
process: each batch needs to be a particular minimum size,
and the coloured products are delivered at a very early stage
in the textile production process.

A process which would allow coloration to take place at a
later stage in the textile production process could offer
much more flexibility. Thus, substantial research has been
undertaken to modify PP fibres with the aim of achieving
improved dyeability from both aqueous- and solvent-based
systems. Different strategies which have been proposed for
modifying PP fibres include:

- direct modification of the constitution of PP fibres
  through the formation of co-polymers in order to
  introduce polar groups directly into the polymer chain,
  which can then serve as sorption sites for the
dyestuff [1,2];
- the incorporation of other polymers (e.g. polyester and
  polymeric additives) leading to the formation of a
  bicomponent fibre with an increased capacity for dye-
  stuff sorption [3–10];
- the incorporation of colourless additives as dye-binding
  sorbents [e.g. minerals, polyhedral oligomeric
  silsesquioxanes (POSS) nanoparticles and clay], which
  also improve the dye-binding capacity [11–14];
- surface modification by plasma activation or the grafting
  of different compounds (e.g. acrylic acid, ethylenedi-
  amine, acrylamide and hexamethyldisiloxane), which
  are used to increase the sorption properties of PP fibres
  [15–18];
- dyeing with selected dyes (although this restricts the
  range to a few colours and shades, and therefore can’t be
  used in wider applications) [19,20].

The use of supercritical carbon dioxide in combination
with selected dyes has also been reported, enabling PP-
dyeing for pale or medium shades [7].

One considerable disadvantage of the incorporation of
substantial amounts of non-PP components during the
process is that they alter those properties particular to PP
fibres; for instance, because of the minimal uptake of
moisture of the hydrophobic fibre to such an extent that
these specific advantages of the fibre disappear.

Many processes for PP-modification are based on applying
solvent-based systems; however, for a textile dyehouse,
a water-based system is preferable, for both ecological and
safety reasons. In this study, a water-based modification of a
PP fibre is presented, one which could be applied using
currently available technical equipment.

The deposition and thermal fixation of cationic PP
dispersions enables a new strategy for modifying the
surfaces of PP fibres; through the presence of cationic

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charges at the fibre surface, the binding of anionic dyes (e.g. acid dyes) can be achieved. In this paper, for the first time, the results of a study to modify the PP fibre surface by the deposition of cationic PP dispersions are presented [21]. A commercial acid dye, CI Acid Red 151, was used as a representative for assessing the dyestuff uptake of the modified fibres. In a systematic investigation, the conditions applied for surface modification were studied with regard to dyestuff-binding capacity and fastness. The modified fibres were also characterised by Fourier Transform-infrared (FTIR) spectroscopy and laser scanning microscopy.

**Experimental**

**Materials**

The materials used for the modification experiments were a knitted PP fabric (99% PP, 1% elastane; mass per area 145 g/m²; Skinflit, Maeder, Austria) and a PP fibre sliver (mass per length 20 g/m; Schoeller Textil, Hard, Austria). To remove the preparation before impregnation, the PP fabric was prewashed in a solution of 1 g/l nonionic detergent (Newalol NL-U; Zschimmer und Schwarz, Mohsdorf, Germany) and 1 g/l anionic detergent (Kieralon B; BASF, Ludwigshafen, Germany) at 40 °C using a commercial household washing machine then dried at ambient room temperature.

**Fibre modification**

Surface modification was achieved by impregnation with a commercial cationic dispersion of maleated PP (Michem Emulsion 28640, 40% solids, pH 4–6; Michelmann, Cincinnati, USA).

For impregnation of the PP samples, an impregnation bath containing cationic PP dispersion with 0, 20, 40, 60, 80 and 100% volumes of dispersion were prepared. The PP samples were then padded with a laboratory paddler (nip pressure 2 bar, 0.5 m/min; Mathis Coating Range CH 8155, Niederhasli, Switzerland). Drying was performed with a laboratory dryer at 100 °C and fixation of the dispersion was performed at 110, 130 or 155 °C (1 min; Mathis Labdryer, Niederhasli, Switzerland).

**Dyeing procedure**

For the dyeing experiments, CI Acid Red 151 (C.I. 26900, Nylosan F-2R) was used. Dyeing experiments were performed for 24 h at an ambient temperature or for 1 h at 60 °C. For dyeing at room temperature, the samples were rinsed three times with cold water then dried at an ambient temperature. For dyeing at 60 °C, the samples were rinsed two times with 60 °C water, once with cold water, then dried at an ambient temperature.

**Colour measurement and photometry**

CIExLab colour coordinates of the samples were determined with a Konica Minolta Spectrophotometer (CM 3610d and Spectra Magic software, d: 8° geometry). The coordinates were calculated for light source D65. L* represents the degree of lightness (0 = perfect black, 100 = perfect white); a* indicates the position on the red-green axis (positive values = red, negative values = green); b* indicates the position on the yellow-blue axis (positive value = yellow, negative value = blue). K/S values were calculated using the Kubelka-Munk equation for the wavelength of maximum absorption.

Photomicrographs of untreated and modified fibres were taken with an optical microscopy (CX41, Olympus, Tokyo, Japan) and a 3D-laser scanning confocal microscope (VK-X200 Keyence, Osaka, Japan).

FTIR spectra of the fibres were recorded in transmission; the spectrum of the dried cationic dispersion was recorded with the use of an attenuated total reflectance (ATR) unit equipped with a diamond crystal using a FTIR spectrometer (Bruker Vector 22 FTIR Spectrometer; spectral range 4000–500 cm⁻¹; resolution 2 cm⁻¹ and 128 scans).

Differential scanning calorimetry (DSC) analysis of the solids in the PP dispersion was taken with a DSC3 Star Mettler-Toledo. For the DSC experiments a sample mass of 12.5 mg was placed in a 100 μl aluminium crucible equipped with a lid. The experimental conditions were as follows: starting temperature: 25 °C; heating rate: 10 °C/min; peak temperature: 200 °C; isotherm: 5 min at 200 °C; cooling rate: 10 °C/min; nitrogen atmosphere: 50 ml/min.

**Fastness measurements**

Wet fastness was determined according to DIN 54006 (change of colour and staining of wool and cotton fabric); wash fastness was determined with a solution of 1.5 g/l fatty alcohol ether sulphate (Newalol NL-U; Zschimmer & Schwarz), 1.05 g/l alkyl poly glycoside (Glucopon EC 650 50%, Henkel, Düsseldorf, Germany), liquor ratio 1:50 at 90–95 °C; isotherm: 5 min at 200 °C; cooling rate: 10 °C/min; nitrogen atmosphere: 50 ml/min.

**Results and Discussion**

The deposition and fixation of the PP dispersion on the PP fibres was achieved through padding, drying and thermofixation. The fixation temperature of the added dispersion must be kept below the melting temperature of the PP fibres. The thermal behaviour of the PP dispersion was studied by DSC. The DSC curves for heating and cooling of the dried PP dispersion are shown in Figure 1.

The endothermal peak for the onset of melting of the PP dispersion starts to develop at a temperature of about 125 °C. Thus, thermal fixation of the dispersion should be achieved above this temperature. During the heating cycle the endothermal peak exhibits a maximum at 158–160 °C, while the onset of the exothermal peak during the cooling cycle is observed at 114–116 °C; the temperature range is dependent on the cooling rate and the presence of nuclei for crystallisation. Therefore, for the thermofixation of the cationic PP dispersion, temperatures of 110, 130 and 155 °C were chosen. Higher temperatures were not used in order to keep loss of orientation in the PP fibres to a minimum, because otherwise this would dramatically alter the physical properties of the fibres.

The modification of the PP fibres through deposition of the dispersions were also studied by FTIR spectroscopy. The infrared spectra of dried PP dispersion, and of
untreated and PP dispersion-treated PP fibres after fixation at 130 °C are shown in Figure 2.

As expected from observing the FTIR spectra of the dried cationic PP dispersion, the major component in the polymer dispersion is PP; only minor absorbance values at 1650 and 1089 cm\(^{-1}\) indicate the presence of other components (e.g. carbonyl groups of the maleated PP dispersion). The concentration of the cationic dispersing agent is below the detection limit of the FTIR technique, and therefore no characteristic signals can be seen in Figure 2b.

Because of the lack in polarity, wetting of a PP surface is extremely difficult, and consequently a good PP dispersion in liquid media is very hard to achieve. By introducing maleic anhydride groups onto PP, usually by free radical grafting, the polarity of PP is increased [23].

The role of maleic anhydride in maleated PP with a higher polarity is to improve the dispersability of PP powder particles in liquid media through cationic surfactants. Also, the wetting of maleated PP particles is improved. During impregnation of PP fibres with cationic PP dispersion by padding, maleated PP powder particles act as carriers for the cationic dispersing agents. In the fixation process, the maleated PP partly melts, adheres to the PP fibre surface, and thereby also fixes the cationic groups. As a result of the higher concentration of charged groups at the fibre surface, a reduction in surface energy is also expected to occur. However, a detailed analysis of this predicted change is beyond the scope of this work.

In the first series of experiments the influence of fixation temperature on dyestuff uptake and fastness of the adsorbed dye was studied. The PP dispersion was padded onto the PP fabric without any further dilution with water, dried, and then thermally treated for 1 min at temperatures of 110, 130 and 155 °C. The take-up of PP dispersion was 105 ± 1.5
wt.% related to the dried sample, and consequently additional PP dispersion of 38.3 ± 0.9 wt.% was achieved.

At the surface of the PP fibre, positively charged sites were introduced as a result of the deposition of the cationic PP dispersion, which then enabled the binding of negatively charged dyestuff molecules. The dyestuff binding mechanism is therefore based on an ion exchange process. This process permits high dyestuff sorption at room temperature. An illustration of the dyeing process is presented in Figure 3.

CI Acid Red 151 was used as a representative dye for the dyeing experiments. The dyestuff absorption was studied in experiments at room temperature and at 60 °C. As the binding of the dyestuff occurs via the formation of ionic bonds between the cationic fibres and acid dye, the dyeing process proceeds at a low temperature. To study the influence of fixation temperature, the PP fabric was thermally treated under the same conditions without the addition of PP dispersion. Photographs of the exhausted dyebaths at the end of the dyeing experiment are shown in the supporting information for this article (Figure S1).

In Table 1, the absorbance of the spent dyebaths at a wavelength of 520 nm, the CIELab coordinates, and the K/S values for the respective dyes (λ<sub>max</sub> = 480 nm) are shown as a function of the temperature applied during thermal fixation and the dyeing temperature. The wavelengths of the maximum absorbance of the dyebaths and the dyes differ slightly because of the influence of the different chemical environments. All the spectrophotometric measurements are therefore given at the respective wavelengths of maximum absorbance. No significant dyestuff absorption was observed at the thermally treated reference samples (1–3 and 7–9).

The low dyestuff sorption on untreated PP samples can be recognised both at the low colour depth of the samples and by the residual high dyestuff content remaining in the dyebath. Dyestuff exhaustion on cationised material reached values higher than 90% of the total dyestuff applied (Figure S2). Lighter dyings, in terms of both CIELab coordinates and K/S, were obtained at a dyeing temperature of 60 °C, which can be explained by the shifting sorption equilibrium and the partial wash-off of weakly bound cationic PP deposits.

The binding of the acid dye is localised to the deposited PP coating, which can also be observed in the respective photomicrographs of dyed samples (Figure 4).

The orange-red coloured parts in the photomicrographs indicate the deposited dyestuff absorbing PP dispersion, with the PP fibres remaining colourless. With a higher fixation temperature, the tendency to form a coating film increases. However, the high amount of PP dispersion reduces the flexibility of the fibres, and the fabric samples appear stiffer and more compact. As can be seen in Figure 4c, the dispersion joins individual fibres and thus reduces the mobility of fibres within the fabric structure.

Photomicrographs obtained by laser scanning microscopy also demonstrate the film formation on modified fibres (Figure 5). For comparison, PP fibres were treated with pure water and heated to 130 °C.

**Fastness properties**

The stability of the surface modification and of the binding between cationic sites and dyestuff was assessed by determining selected colour fastness properties. In this study, the wet fastness and the wash fastness at 40 °C were used as indicators for dyestuff binding. The results for wash fastness (Table 2) indicate significantly higher fastness for dyeings obtained with a dyebath temperature of 60 °C.

![Figure 3: Reactions for sorption of CI Acid Red 151 on the binding sites of the modified fibre surface.](image-url)

| Sample | Additional PP dispersion, wt.% | Fixation temperature, °C | Absorbance, 520 nm | L* | a* | b* | K/S, 480 nm |
|--------|--------------------------------|--------------------------|-------------------|----|----|----|-------------|
|        |                                |                          |                   |    |    |    |             |
| RT     |                                |                          |                   |    |    |    |             |
| 1      | –                              | 110                      | >3.0              | 69.6 ± 0.3 | 13.6 ± 2.0 | 6.7 ± 1.1 | 0.8 ± 0.0  |
| 2      | –                              | 130                      | >3.0              | 69.3 ± 1.1 | 13.4 ± 0.8 | 6.7 ± 1.1 | 0.9 ± 0.1  |
| 3      | –                              | 155                      | >3.0              | 69.2 ± 1.0 | 13.8 ± 0.0 | 8.8 ± 1.0 | 0.9 ± 0.0  |
| 4      | 38.3 ± 0.9                     | 110                      | 0.10 ± 0.13       | 45.1 ± 2.0 | 45.8 ± 0.9 | 26.5 ± 1.7 | 9.4 ± 0.4  |
| 5      | 38.3 ± 0.9                     | 130                      | 0.09 ± 0.01       | 45.0 ± 0.5 | 45.5 ± 0.9 | 25.4 ± 0.4 | 9.2 ± 0.3  |
| 6      | 38.3 ± 0.9                     | 155                      | 0.42 ± 0.07       | 45.0 ± 1.5 | 42.7 ± 0.7 | 22.6 ± 1.1 | 8.7 ± 1.4  |
| 7      |                                |                          |                   |    |    |    |             |
| 8      |                                |                          |                   |    |    |    |             |
| 9      |                                |                          |                   |    |    |    |             |
| 10     | 38.3 ± 0.9                     | 110                      | 1.02 ± 0.11       | 49.1 ± 0.5 | 44.3 ± 1.5 | 23.1 ± 1.74 | 7.2 ± 0.3  |
| 11     | 38.3 ± 0.9                     | 130                      | 0.71 ± 0.11       | 47.9 ± 0.5 | 46.7 ± 0.3 | 24.5 ± 0.8 | 7.7 ± 0.7  |
| 12     | 38.3 ± 0.9                     | 155                      | 0.40 ± 0.11       | 48.3 ± 1.3 | 43.9 ± 0.4 | 21.1 ± 1.4 | 7.1 ± 0.2  |

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Colour change, and staining of wool and cotton fabric in the wash fastness tests were rated 3.5–4, which corresponds to an acceptable level of fastness. Lower fastness results were determined for dyeing at room temperature, which can be explained both by the release of weakly bound parts of the coating, and the desorption of dyestuff molecules. This finding is in agreement with the lighter dyeings obtained at a dyeing temperature of 60 °C.

Values for colour change in wet fastness tests were 4–4.5. Marks for the staining of wool and cotton samples in wet fastness tests increased with fixation temperature and reached values of 3.5 after fixation at 155 °C.

In the first series of experiments, higher amounts of PP dispersion were used to study dyestuff uptake and fastness. No saturation effects with regard to the capacity of cationic binding sites on the modified PP fibres were observed. In the second series of experiments, the amount of cationic PP dispersion was reduced in order to evaluate limiting the conditions for dyestuff uptake and colour depth. The amount of PP dispersion applied by padding was reduced stepwise from 100 to 0% volume. Fixation was performed at 110 and 130 °C. The dependence of colour depth on the amount of PP dispersion padded onto the fabric is shown in Figure 6. The experimental conditions, colour depth with CI Acid Red 151 in terms of CIELab coordinates, and K/S values can be seen in the supporting information for this article (Table S1). For comparison, the colour coordinates of selected undyed samples are also shown.

A relationship between the amount of PP dispersion applied and dye adsorption is observed in Figure 6. K/S values increase and the L* coordinates decrease with increasing amounts of PP dispersion; however, the non-linear dependency of L* on dyestuff concentration resulted in relatively small changes of L* when more than 40% volume of PP dispersion was used in the padding liquid. This result is also reflected by the dyestuff exhaustion, which increases with the content of cationic PP dispersion.

In Figure 7, the photometric absorption curves of exhausted dyebaths and the absorbance at 500 nm are shown. A fixation temperature of 130 °C had been applied to these samples.

The absorption curves obtained by photometry of the spent dyebaths are in agreement with the CIELab coordinates and K/S values of the dyed samples. For unmodified PP, only staining was observed, while substantial absorption had already been obtained at a concentration of 20% volume of cationic PP dispersion in the padding liquid. Almost complete exhaustion is observed above 40% volume of PP dispersion, which is in agreement with the results of the wash fastness tests in terms of K/S.
Table 2 Wash fastness properties of modified and untreated CI Acid Red 151 dyed PP fabric samples (colour change and staining of wool and cotton)

| Sample  | PP dispersion, wt.% | Temperature, °C | Colour change | Staining wool | Staining cotton |
|---------|---------------------|-----------------|---------------|---------------|----------------|
| RT      | 4                   | 110             | 3             | 4.5           | 3              |
| 2       | 38.3 ± 0.9          | 110             | 2.5–3         | 4             | 2              |
| 3       | 38.3 ± 0.9          | 155             | 3             | 3.5           | 2              |
| 4       | 38.3 ± 0.9          | 110             | 4             | 3.5–4         | 4              |
| 5       | 38.3 ± 0.9          | 130             | 4             | 3.4–4         | 3.5–4          |
| 6       | 38.3 ± 0.9          | 155             | 4             | 4             | 4              |
| 7       | 38.3 ± 0.9          | 130             | 3.5–4         | 4             | 3.5–4          |
| 8       | 38.3 ± 0.9          | 110             | 3             | 3.5           | 3.5–4          |
| 9       | 38.3 ± 0.9          | 155             | 4             | 4             | 3.5–4          |
| 10      | 38.3 ± 0.9          | 110             | 4             | 3.5–4         | 3.5–4          |
| 11      | 38.3 ± 0.9          | 130             | 4             | 4             | 3.5–4          |
| 12      | 38.3 ± 0.9          | 155             | 4             | 4             | 3.5–4          |

Figure 6 Colour depth of dyed samples in terms of L* and K/S (500 nm) as a function of PP dispersion padded onto the fabric (fixation at 110 and 130 °C)

Figure 7 Absorption of exhausted dyebaths as a function of the amount of PP dispersion: (a) initial absorption of dyebath and absorption curves of spent dyebaths; and (b) absorption at 500 nm as a function of the amount of PP dispersion (samples 19–25 are given in Table S2); values for absorption >2 were calculated from measurements made at diluted concentrations of solution.
fixed by ionic bonding). The fibre core still consists of 100% PP. In the case of CI Acid Red 151, fixation proceeds at room temperature and the dyeing of dark shades is possible. Wash fastness and water fastness were found to be at acceptable levels with values of 3.5–4.5, indicating a stable binding of the acid dye. Untreated PP fibres exhibited only minor staining with CI Acid Red 151.

Microscopic investigations indicated the formation of a surface film. At a fixation temperature of 110 °C, the surface adhesion of the film is limited and a substantial release of the coating during mechanical processing was observed. The release of particulate material is substantially reduced at higher fixation temperatures of 130 and 155 °C.

Almost complete dyebath exhaustion was observed within 24 h at room temperature, which suggests a possibility of saving energy when dyeing at low temperatures. A reduction in the applied amount of cationic PP dispersion led to a gradual reduction in dyestuff uptake.

The results demonstrated the applicability of the technique on PP fibres, both in the form of fibre bands and as knitted fabric.

This new fibre modification enables the PP-dyeing step to be introduced at a later stage of the fabric production process, and it also permits the preparation of fibre blends (e.g., with wool), which can then be dyed in the same dyebath. For instance, wool/PP fibre blends are used in functional sports clothing.

Compared to the mass coloration of PP fibres, the process will be more expensive, as additional chemicals and more processing steps (padding, drying and fixation) are required. However, the technique permits a substantially higher flexibility, both in choices of colour and with regard to the minimum amount of material which has to be dyed.

The film-forming properties of the coating led to a stiffer handle of treated samples; this will require optimisation in future studies, for instance, through the use of PP dispersions with a lower melting point, a higher content of cationic charges in the dispersion, and the application of appropriate mechanical processes to restore the desired fabric softness. Future research should therefore focus on the characterisation of fibre properties, which will be critical for successfully scaling up technical aspects of the process, including:

- determination of colour depth and fastness properties for a useful colouristic range of acid dyes;
- fibre adhesion due to the presence of PP dispersion and the consequences for yarn spinning;
- yarn friction and dust development through abrasion of particulate PP material (e.g., at yarn brakes and needles in knitting);
- modification of the PP dispersion with regard to melting range;
- application of the PP dispersion to different stages of textile production (fibre, yarn, fabric and printing techniques) and effects on fabric handle and softness.

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### Supporting Information

Additional Supporting Information may be found online in the Supporting Information section at the end of the article:

**Figure S1** Photographs of dyeing experiments at room temperature. (a) PP-dispersion treated samples (temperature
of fixation 110, 130, 155 °C) and (b) unmodified reference samples

**Figure S2** Photograph of the dyed sample 11. Impregnation with 100% PP dispersion, fixation at 130 °C.

**Table S1** Fabric modification with reduced amounts of PP-dispersion.

**Table S2** Fibre band modification with different amounts of PP-dispersion.