PLASMA IN TEXTILE TREATMENT

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Introduction

Plasma modification of textiles represents great opportunity for improvement of older, energetically demanding, slow and sometimes not very eco-friendly treatment technologies. Application of plasma is ecological and friendly for production costs due to energy savings and reduction of processing times. Compared to other scientific works describing increase of hydrophilicity of hemp, wool or polypropylene textiles or decrease of felting shrinkage of wool[1,2], our activities were focused mainly on effects important for manufacturers and their interest in plasma applications as a new advantageous technology.

In this work we present possibilities of utilizing plasma pre-treatment in three different textile-processing areas. At first, the possibility of dyeing of plasma pre-treated cotton at temperatures lower as usual was tested. In the second experiment, the recent method of textile printing – inkjet print, normally suitable mainly for cotton substrates, was tested on very hard-to-print polypropylene with plasma pre-treatment. Finally the last experiment proved that plasma is capable to photo catalytically activate TiO_2, applied on textile substrate, which is subsequently able to inhibit bacteria growth on surface.

The main reason for using plasma pre-treatment is saving of quite considerable amounts of energy during dyeing process. Normally, cellulose textiles like cotton have to be heated to 100 °C and this level has to be held for ca 30 min. Hydrophilicity increased by plasma should lead to good results using lower dyeing temperatures and/or shorter processing times. Therefore interesting power savings are possible.

Polypropylene is known as very hard-to-print and hard-to-dye material. Use of conventional printing technologies is difficult and limited.

Inkjet printing is novel method of textile printing, utilizing printers similar to office inkjet ones. In the present, these types of printers are capable to print only onto hydrophilic substrates, i.e. textiles with high content of cotton. Plasma pre-treatment of polypropylene should increase colourfastness of inks and their better fixation on polypropylene fibres.

Photocatalytic properties of TiO_2 activated by UVA irradiation or by visible light are well known[3]. In presented experiment, the activation of TiO_2 by plasma is studied and expressed in unconventional manner – by antibacterial activity of textile sample with colloidal TiO_2 application. Antibacterial attributes were tested according to ASTM E2149-1 standard.

In all experiments the comparison between plasma treated and untreated samples was made in order to show effect of plasma modification.

Experimental

All experiments were carried on using laboratory coplanar low temperature atmospheric plasma discharge with power of 400 W. Exposition time was in all cases 5 s. Samples were processed immediately after plasma pre-treatment (except of inkjet printing experiments, where transport to printer took approximately 30 minutes).

For experiments with dyeing white woven cotton fabric was used. As a dye, industrial saturn navy blue was used. Samples were processed in laboratory dyeing device AHIBA. Washing of samples was performed in laboratory washing machine.

Inkjet prints were performed on knitted, 100% polypropylene white t-shirt (producer Klimatex, Czech Republic), utilizing standard small inkjet printer and commercial inks, used in external graphic studio. Washing of samples was performed in laboratory washing machine Wascator FOM71MP-Lab.

TiO_2 (anatase) nanoform colloidal solution (containing 0,8 % of TiO_2) was purchased from TitanPE Ltd., China. *Klebsiella Pneumoniae* (Czech Collection of Microorganisms strain) was used for antibacterial testing.

For determination of colour differences between plasma-treated and untreated samples EN ISO 105-A02 was used. Domestic laundering was carried-on according to standard EN ISO 6330.

Differences between inkjet-printed samples before/after home laundering were specified by eye observation.

Determination of antibacterial activities of plasma-treated/untreated samples was carried on according to ASTM E2149-1 standard.

1.) Dyeing of plasma pre-treated cotton woven fabrics:

Three different temperature modes were used for dyeing process (see Fig. 1):

– mode A: maximal temperature of 100 °C
– mode B: maximal temperature of 80 °C
– mode C: maximal temperature of 60 °C

Two samples (woven cotton fabric, weight 10 g, dimension ca 28×23 cm) were prepared for every temperature

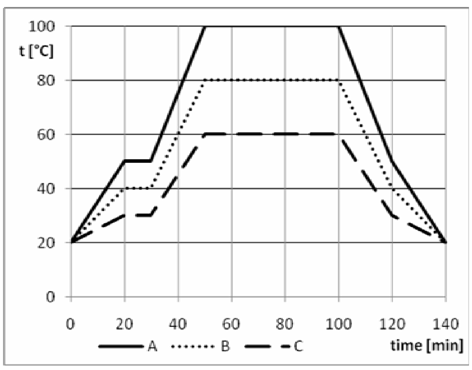

Fig. 1. **Temperature modes of dyeing**

mode; first one without plasma pre-treatment, second one with plasma pre-treatment (in Fig. 3–6 marked with *). Dyeing bath (distilled water) volume for every sample was 400 ml, with 0,05 g of Saturn navy blue dye itself. Dyeing was carried out using laboratory dyeing apparatus AHIBA NUANCE Top Speed II.

Plasma pre-treated samples were inserted into dyeing bath and dyed immediately after plasma surface modification (400 W, exposition time 5 s). After dyeing, all samples were dried at room temperature.

Colour fastness of samples was evaluated after five washing cycles using standard washing machine WASCATOR FOM71MP-Lab (30 °C, 3 g l^{-1} of ECE detergent).

2.) Inkjet print on plasma pre-treated polypropylene knitted fabric:

For this experiment two samples were prepared; with and without plasma pre-treatment. Plasma modified sample was exposed to plasma ca 30 minutes prior to the main print. The same test picture (see Fig. 2.), consisting of different print elements like photo, text, vector graphic etc. was printed on both samples.

After printing, dyes were thermally fixed on both samples for 30 seconds under 100 °C.

Colour fastness of samples was evaluated after five washing cycles using standard washing machine WASCATOR FOM71MP-Lab (30 °C, 3 g l^{-1} of ECE detergent).

3.) Photocatalytic activation of TiO_2 on textile by means of plasma:

Two cotton woven samples were prepared; first without plasma activation and second by plasma pre-treated. Colloidal TiO_2 was applied on samples using laboratory dyeing device AHIBA NUANCE Top Speed II. Samples were rinsed and then for 1 minute dried using 160 °C iron. Afterwards, first sample was exposed to plasma (exposition time 5 s), while second wasn't. Both samples were tested for antibacterial activity according to ASTM E2149-1 standard. Difference in antibacterial activity of plasma-treated and untreated sample as well as duration of antibacterial effect over time were checked.

According this standard nutrient broth with specified concentration of selected bacteria (*Klebsiella Pneumoniae*) was prepared. Then the specified weight of sample was inserted into broth solution and agitated. After desired times small amount of solution was taken and poured into agar plates. On the plate, colonies of microorganisms were grown after specified time and then counted (CFU – colony forming unit). High number of CFU represents small antibacterial effect of sample. For comparison, the "blank" broth solution without any sample (represents untreated textile sample) was processed in the same way. Results are expressed as proportional reduction of bacteria on tested sample against this blank sample.

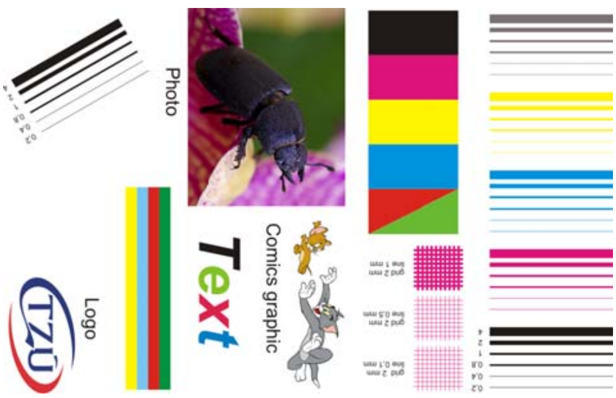

Fig. 2. **Test picture for inkjet print**

Results and discussions

1.) Dyeing of plasma pre-treated cotton woven fabrics

Experimental results of dyeing are in Fig. 3. After all three dyeing modes the plasma pre-treated samples show brighter and deeper colour shade. Fig. 4–6 show appearance of samples after first, third and fifth washing cycle, respectively.

Difference between treated an untreated samples is clearly visible. Plasma pre-treated samples show deeper colour shade than untreated samples. Biggest differences can be seen after dyeing. After several washing cycles the difference decreases. It means that used plasma helps the textile to absorb more dye from dyeing bath, but has only small impact on colourfastness of textile material (colourfastness is in this case more significant than dyeing technology).

Samples with plasma treatment are marked " * ".

2.) Inkjet print on plasma pre-treated polypropylene woven fabric

Results before and after two washing cycles are presented in Fig. 7 and 8, respectively.

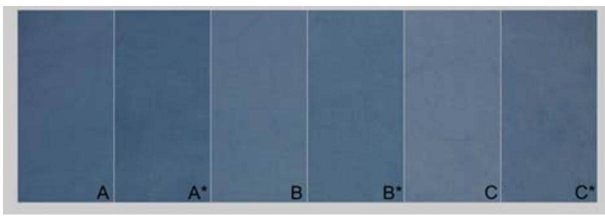

Fig. 3. **Samples after dyeing**

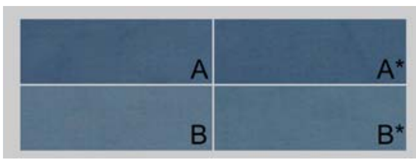

Fig. 4. **Samples after first washing cycle**

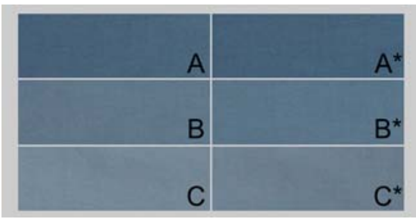

Fig. 5. **Samples after third washing cycle**

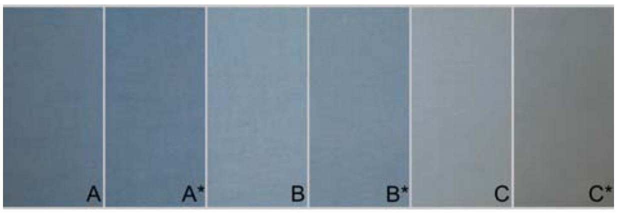

Fig. 6. **Samples after fifth washing cycle**

no plasma plasma pre-treatment

Fig. 7. **Inkjet prints on PP before washing**

Even before washing, the plasma pre-treated sample show much brighter colour shades. After two washing cycles the difference between treated and untreated sample is obvious. It means that plasma is able to bring added value to inkjet printing on polypropylene so that it participates on hydrophilization of substrate and partially in sorption of inks on samples. Results are very positive and this topic will be subject of further research.

Table I summarizes numerical expression of colour fastness results according to EN ISO 105-A02 standard, measured on colour rectangles, positioned above the photograph in tested picture. In four from six measurements treated sample exhibits better colourfastness than untreated.

no plasma plasma pre-treatment

Fig. 8. **Inkjet prints on PP after two washing cycles**

Table I
Colourfastness level of samples (1- bad colourfastness, 5- very good colourfastness)

Colour in sample	Treated sample	Untreated sample
black	4	2
violet	3	4
yellow	2–3	1–2
blue	2	1–2
red	4	1
green	2–3	2–3

Table II
Antibacterial activity of TiO_2 doped sample in time

Time [hours]	Tested sample [CFU]	“Blank” sample [CFU]	Reduction [%]
0	86	262	67
1	4	148	97
2	6	172	96
3	38	237	84
5	153	400	62

3.) Photo catalytic activation of TiO_2 on textile by means of plasma

Results of antibacterial tests are summarized in Table II. Time dependence of proportional reduction is shown in Fig. 9.

Results show good antibacterial effect and confirm that plasma is able to activate TiO_2 (note: TiO_2 without activation has no antibacterial, nor photocatalytical properties) for antibacterial effectiveness. Activated TiO_2 can inhibit bacteria

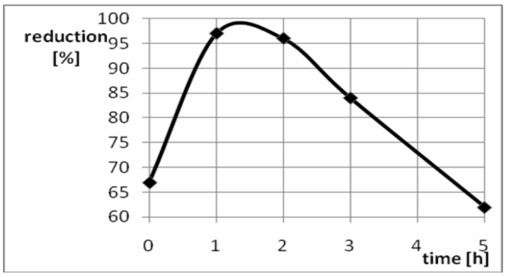

Fig. 9. **Reduction of *Klebsiella Pneumoniae* in time**

growth more then 5 hours after activation, even if it's placed in dark.

Conclusion

All three experiments showed more or less positive results, proving effect of plasma treatment on processes of dyeing and printing as well as ability to photo catalytically activate TiO_2 on textile substrate in very short time. All those effects should lead to innovative and energy-saving modifications of traditional textile processing. Novel technologies mainly for inkjet textile printing and photo catalytic activation of TiO_2 can extend nowadays possibilities and help producers to offer new or innovative products. All three reported themes are subject to further research.

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P. Nasadil* and P. Benešovský *(Textile Testing Institute, Brno, Czech Republic):* **Plasma in Textile Treatment**

The paper deals with three different initial experiments focused on treatment of different textile materials (polypropylene and cotton) by low temperature atmospheric air plasma and its effects. Main idea was to prove positive influence of plasma applications to manufacturers and increase of market value of textile articles by improved dyeing, affinity to prints and antimicrobial effect.

Modified parameters of textiles were tested according to EN standards as standardized tests are important and known by textile producers and also by end-users of textile products. Change of colour characteristics and dyeing effects after plasma pre-treatment, effects of plasma on appearance of inkjet prints and photo activation effect of plasma on TiO_2 treated cotton – as ecological antibacterial activity are reported.