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APPLICATION OF FLAME RETARDANT MICROCAPSULES TO POLYESTER AND COTTON FABRICS

NANOS MIKROKAPSUL Z ZAVIRALCEM GORENJA NA POLIESTRNO IN BOMBAŽNO BLAGO

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A microencapsulated fire retardant was used with the intention to produce fire retardant textiles. Melamine-formaldehyde polymer-wall microcapsules with a triphenyl phosphate core were applied to both a cotton woven and a polyester nonwoven fabric using impregnation and screen printing. The samples were observed with a scanning electron microscope (SEM). The combustion performance of the textile samples was examined with the limiting oxygen index (LOI) and the vertical burning test. The thermal properties of the microcapsules and fabrics were examined using TGA and DSC analyses. The mass per unit area, rigidity and air permeability of the treated fabrics were tested. The results show that the microcapsules with triphenyl phosphate can be successfully applied to cotton and polyester fabrics using screen printing and impregnation methods, but the fire retardation was successful only at the highest concentration of the microcapsules. At this concentration, the mechanical properties of the starting materials appear to deteriorate.

Keywords: polyester, cotton, textile, microcapsules, fire retardant, triphenyl phosphate

V raziskavi je bil uporabljen mikrokapsuliran zaviralec ognja z namenom ustvariti ognjevarno tekstilijo. Mikrokapsule z melamin-formaldehidno ovojnico in trifenil fosfatnim jedrom so bile uporabljene na bombažni tkanini in poliestrni vlaknovini z impregnacijo in filmskim tiskom. Vzorci so bili pregledani z elektronskim mikroskopom (SEM). Gorenje tekstilnih vzorcev je bilo preučeno z vrednostjo kisikovega indeksa (LOI) in vertikalnega preizkusa gorenja. Termične lastnosti mikrokapsul in blaga so bile preiskane z uporabo TGA- in DSC-analize. Pri obdelanih vzorcih je bila izmerjena ploščinska masa, togost in zračna prepustnost. Iz rezultatov je razvidno, da se da mikrokapsule s trifenil fosfatom uspešno aplicirati na bombaž in poliester z impregniranjem in tiskanjem, vendar pa je bilo zaviranje gorenja uspešno le pri najvišji koncentraciji mikrokapsul. Pri tej koncentraciji pa se mehanske lastnosti začetnih materialov očitno poslabšajo.

Ključne besede: poliester, bombaž, tekstil, mikrokapsule, zaviralec gorenja, trifenil fosfat

1 INTRODUCTION

There are many factors that influence the flammability of textiles. These factors are the composition of fibres; the construction of the yarn; the presence of certain finishes, dyes, impurities or detergent residues; the geometry or position of the material; the temperature; the relative humidity; the air flow; and the presence of oxygen. Various fibres react differently to a flame exposure.

To guarantee human safety, textiles can be treated with different fire retardants (FR). These are chemicals that reduce the flammability of the material to which they are applied.[1] Fire-retardant materials do not burn; however, these materials show certain physical and chemical changes after the removal of a flame source.[2]

Synthetic fibres may be rendered flame retardant during their production, thereby creating a degree of inherent flame retardancy. The retardant additives can be incorporated in the polymer melt/solution prior to extrusion or retardant molecules can be grafted onto the main polymeric chain.[3] The other way of protecting synthetic fibres, and the only way of protecting natural fibres, from burning is by applying suitable flame retardant finishes. The best and the most durable FRs for cellulose are those based on phosphorus (P) and nitrogen (N) that can react with the fibres or create a cross-linked structure on the fibres. Phosphorus and/or halogen compounds are among the fire retardants that confer good protection to polyester. Phosphorus compounds are therefore effective for use with both cotton and polyester fibres.[4] One such compound is triphenyl phosphate (TPP) (**Figure 1**).

TPP is widely used as a non-solvent plasticiser for cellulose acetate films, giving flexibility and toughness to the films; it is also an excellent FR agent and plasticiser for synthetic resins based on phenolics and phenylene oxide as well as formaldehyde in the production of

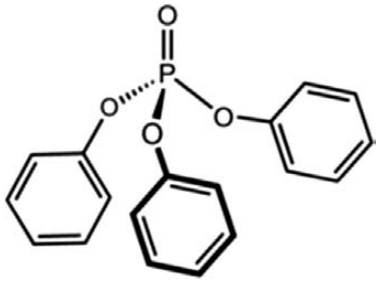

Figure 1: Structure of triphenyl phosphate
Slika 1: Struktura trifenil fosfata

stencil blanks, dopes, films, varnishes, plastics, lacquers, etc.[5] TPP breaks down in the flame to produce chemical species such as P_2, PO, PO_2 and HPO_2. These reactions reduce the hydrogen atom concentration in the vapour phase, thus extinguishing the flame.[6]

The techniques used for the flame retardant finishing of fabrics are mostly padding and coating. The disadvantages of applying retardants this way include the stiffness of the treated fabrics and negative effects on the colouring. One way to resolve this problem may be the use of microencapsulation. Microencapsulation is defined as "a technology of packaging solids, liquids or gaseous materials in miniature sealed capsules that can release (or not) their contents at controlled rates under the influence of specific conditions."[7] In this way, the active compounds are safely stored inside the capsules, isolated from their surroundings, and they are protected from any degrading factors.[8–11] In the last few years, there have been reports of applying microcapsules loaded with a fire retardant to textiles to provide a reliable protection from burning. Di-ammonium hydrogen phosphate (DAPH) is the compound used most often as a FR in such researches.[12–16] However, no paper describing the use of encapsulated TPP to protect textiles from burning was found.

The goal of our work was to prepare microcapsules with a triphenyl phosphate core and melamine-formaldehyde wall using the *in-situ* polymerisation method and to apply these microcapsules to cotton woven (CO) and polyester nonwoven fabrics (PES) (both are highly flammable) using screen printing and impregnation methods. The objective of the research was to determine whether the chosen fire retardant acts successfully on these two materials when encapsulated and whether the printing and impregnation methods of the microcapsule application provide a good fire retardant protection. The intention was to determine the optimum concentration of the microcapsules in the printing paste and in the impregnation bath. The flame retardancy of the samples was determined with a vertical burning test and a LOI analysis. The thermal behaviours of the microcapsules and fabrics were examined with thermogravimetric (TGA) and differential scanning calorimetry (DSC) analyses. The distribution, dimensions and form of the microcapsules on the printed and impregnated fabrics were observed using SEM. The fabric properties of the treated materials were tested with standardised methods.

2 EXPERIMENTAL WORK

2.1 Material

A bleached and mercerised 100 % cotton woven fabric (124 g/m^2 in mass, supplied by Tekstina, d. d., Slovenia) and a 100 % polyester nonwoven fabric (184 g/m^2 in mass, obtained from Filc, d. d., Škofja Loka, Slovenia) were used for the study. A suspension of the microcapsules (MC) with a size of 1–7 μm, a pressure-sensitive melamine-formaldehyde wall and a solid triphenyl phosphate core was prepared with an *in-situ* polymerisation of melamine-aldehyde prepolymers.[17] The suspension was added to the printing paste, composed of a synthetic polyacrylate thickener, Tubivis DRL 300, and a polyacrylate binder, Tubifast AS 30, both obtained from CHT, Germany. Padding binder FM/N (acrylic polymer solution from Minerva, Italy), in combination with an ammonium sulphate catalyst (Sigma Aldrich, Germany), dispersing agent Sinergil BT/N (Minerva, Italy) and an MC suspension, was used in the preparation of the impregnating baths.

2.2 Printing and impregnation

Table 1 presents the recipes for four printing pastes with an increasing quantity of MCs. Paste P4 was prepared with the highest concentration of microcapsules (600 g/kg) that still allowed its formulation. It was not possible to prepare a more concentrated paste due to the other required ingredients. The printing, drying and curing conditions are presented in **Table 2**.

Table 1: Printing paste recipes
Tabela 1: Recepture tiskarskih past

Component	Quantity (g/kg)			
	P1	P2	P3	P4
Polyacrylate thickener	34	34	34	34
Polyacrylate binder	150	150	150	150
MCs	0	200	400	600
Distilled water	816	616	416	216

Table 2: Printing, drying and curing conditions for the flat screen printing
Tabela 2: Razmere pri tiskanju, sušenju in fiksiranju pri ploskem filmskem tisku

Phase	Condition
Printing	Flat screen stencil: mesh of 43 threads/cm Printing speed: 80 % Squeegee diameter: 8 mm Magnet pressure: level 5 No. of passes: 2
Drying	Air drying
Thermal curing	Ernst Benz TKF 15-M500 drier, T = 150 °C, t = 5 min

Flat screen printing was performed on a laboratory magnetic printing machine, MINI MDF R 390, Johannes Zimmer AG (Austria). The area coverage of the printing paste on the cotton and felt cloth was approximately 25 cm × 35 cm.

Impregnation baths with an increasing quantity of MCs were prepared according to **Table 3**. Impregnation bath I4 was prepared with the maximum possible concentration of microcapsules (800 g/kg), which means that no distilled water was added. It was not possible to prepare a more concentrated bath due to the other required ingredients. The impregnation, drying and curing conditions are presented in **Table 4**.

The impregnation was performed on the Mathis 2-roll horizontal laboratory foulard (Switzerland).

Table 3: Impregnation bath formulas
Tabela 3: Recepture impregnirnih kopeli

Component	Quantity (g/kg)			
	I1	I2	I3	I4
Binder	140	140	140	140
Catalyst (water-to-catalyst ratio 2:1)	10	10	10	10
Dispersing agent	50	50	50	50
MCs	0	200	400	800
Distilled water	800	600	400	0

Table 4: Impregnation, drying and curing conditions
Tabela 4: Razmere pri impregniranju, sušenju in fiksiranju

Phase	Condition
Padding	Wet pick up of 100 % – cotton fabric Wet pick up of 330 % – polyester felt No. of passes between rolls: 1
Drying	Air drying
Thermal curing	Ernst Benz TKF 15-M500 drier, T = 150 °C, t = 3 min

The abbreviations of all the samples used in this study are listed in **Table 5**.

Table 5: Abbreviations of the samples
Tabela 5: Okrajšave oznake vzorcev

Abbr.	Sample	Abbr.	Sample
PES	PES fabric – untreated	CO	CO fabric – untreated
PESp0	PES printed without MC	COp0	CO printed without MC
PESi0	PES impregnated without MC	COi0	CO impregnated without MC
PESp200	PES printed with 200 g/kg MC	COp200	CO printed with 200 g/kg MC
PESi200	PES impreg. with 200 g/kg MC	COi200	CO impreg. with 200 g/kg MC
PESp400	PES printed with 400 g/kg MC	COp400	CO printed with 400 g/kg MC
PESi400	PES impreg. with 400 g/kg MC	COi400	CO impreg. with 400 g/kg MC
PESp600	PES printed with 600 g/kg MC	COp600	CO printed with 600 g/kg MC
PESi800	PES impreg. with 800 g/kg MC	COi800	CO impreg. with 800 g/kg MC

2.3 Analysis

2.3.1 Washing

The samples were laundered for 30 min at 40 °C according to the ISO 105-C01:1989 (E) standard, using a soap solution (5 g/l of soap) with pH 7 and a liquor-to-fabric ratio of 50 : 1.

2.3.2 SEM observations

The uniformity of the deposit, size and morphological characteristics of MCs on the finished fabrics were observed with a scanning electron microscope (Jeol JSM 606). The samples were coated with gold.

2.3.3 Combustion test

The combustion performance was studied with the LOI and the vertical burning test. The LOI was measured using a limiting oxygen index chamber (Dynisco, USA) according to standard ASTM D 2863. The vertical burning test was performed in the burning chamber according to the DIN 53906 standard.

2.3.4 Thermal properties of microcapsules and the fabrics treated with microcapsules – TGA and DSC analyses

The samples were examined with a 449c Jupiter instrument (NETZSCH). The samples were placed on Al_2O_3 carriers. They were heated in a protective atmosphere (air) and the measurements were performed from 35–650 °C at a heating rate of 10 K/min. The samples were then cooled at 10 K/min to room temperature.

2.3.5 Fabric properties

The fabric mass per unit area was determined according to the SIST EN 12127:1999 standard, and the fabric air permeability was determined according to the SIST EN ISO 9237:1999 standard.

3 RESULTS AND DISCUSSION

3.1 SEM micrographs

There are differences in the structure between the cotton woven fabric and polyester nonwoven fabric because of different ways, in which they are made. The cotton woven fabric is made in a weaving process, where the yarns are packed close together with high forces and the fabric is therefore thin, smooth and compact, with the structure in good order. The polyester nonwoven fabric is made in a process of needle-punching directly from the fibres, with less tension during the production and it is therefore thicker, more porous and hairy, having a disordered structure. These structural differences influence the absorption and retention of added substances.

The micrographs in **Figure 2** present the impregnated and printed cotton and polyester samples. Many microcapsules are present on the fabrics; they are round in shape and not damaged. The microcapsules are more evenly distributed on the woven CO samples (**Figures**

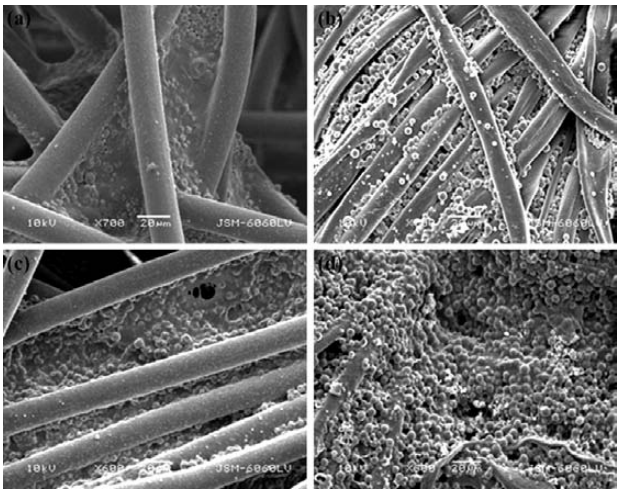

Figure 2: SEM micrographs of: a) PESi400, b) COi400, c) PESp400 and d) COp400

Slika 2: SEM-posnetki vzorcev: a) PESi400, b) COi400, c) PESp400 in d) COp400

2b and **d**) than on the nonwoven PES samples (**Figures 2a** and **c**). Because of the porous structure of the nonwoven PES, the microcapsules are captured in the nooks among the fibres and only a few microcapsules are located on the fibres. The printed CO sample (**Figure 2d**) is almost completely covered with microcapsules, while the impregnated CO sample has fewer microcapsules on the surface (**Figure 2b**). This effect is due to the printing process (the paste was applied onto the fabric horizontally with a squeegee) and the rheological properties of the paste (thixotropy), which prevented the capsules passage through the yarn during printing. Impregnating, on the other hand, is a mechanical process that allows the products to pass through a yarn, so it seems that most of the microcapsules are located in the inner part of the yarn of the impregnated fabric.

3.2 Combustion tests (the vertical burning test and LOI)

The results of the vertical burning test and LOI are shown in **Tables 6** and **7**, respectively. **Figure 3** represents the untreated and treated PES and CO samples after the burning test.

The upward burning behaviour shows that the PES samples burn longer than the CO samples and do not glow, whereas the CO samples glow longer than they burn. An addition of TPP microcapsules prolongs the burning time of the PES samples, while it has no influence on the burning time of the cotton samples with less than 800 g/kg of microcapsules. The samples of cotton burned through their whole length, so that there was no residual cloth left, only some ash. In the case of PES, the flame did not spread over the whole length of the sample, so that there was some unburned fabric. Neither material burned when impregnated with 800 g/kg of microcapsules (PESi800, COi800), as they were both self-extinguishing (**Figure 3**). These samples were tested for the LOI. Both had an LOI of 25, meaning that the ignition and burning were slowed down. The LOI should be at least 26 to consider the samples to be flame retardant. The washed samples had a lower LOI of 24, most likely due to the removal of some microcapsules during the washing process.

Table 6: The results of the vertical burning test of the printed and impregnated samples with different quantities of the applied microcapsules

Tabela 6: Rezultati vertikalnega preizkusa gorljivosti tiskanih in impregniranih vzorcev z različno količino nanesenih mikrokapsul

Sample	Burning time (s)		Glow time (s)	
	unwashed	washed	unwashed	washed
PES	14	15	0	0
PESp0	50	44	0	0
PESi0	44	33	0	0
PESp200	90	114	0	0
PESi200	50	43	0	0
PESp400	80	117	0	0
PESi400	50	37	0	0
PESp600	50	33	0	0
PESi800	0	5	0	0
CO	12	9	20	7
COp0	5	3	20	30
COi0	7	6	25	20
COp200	5	5	40	44
COi200	8	9	20	10
COp400	19	9	9	15
COi400	16	11	3	8
COp600	15	10	10	13
COi800	0	9	0	0

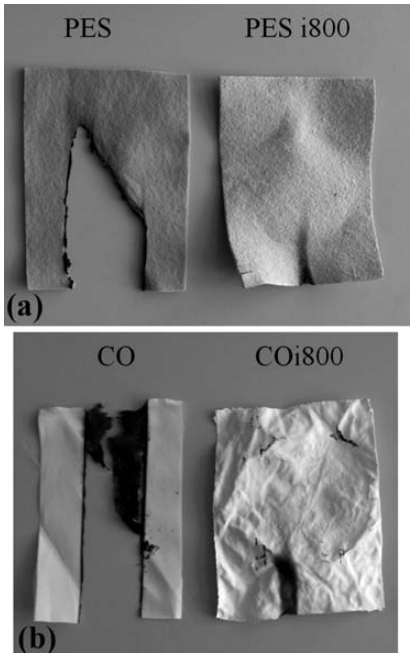

Figure 3: Samples of the untreated and impregnated fabrics after the vertical burning test: a) PES and b) CO

Slika 3: Neobdelani in impregnirani vzorci po vertikalnem preizkusu gorenja: a) PES in b) CO

Table 7: Results of the LOI test of the unwashed and washed samples impregnated with the maximum concentration of microcapsules
Tabela 7: Rezultati preizkusa LOI neopranih in opranih vzorcev, impregniranih z najvišjo koncentracijo mikrokapsul

Sample	LOI (%)	Time of burning (s)
PESi800	25	180
PESi800 washed	24	120
COi800	25	60
COi800 washed	24	70

3.3 TGA and DSC analyses

Figures 4 and **5** represent the TGA and DSC curves of the suspensions of the microcapsules with and without a TPP core, composed only of melamine resin (a blend). The TGA curves reveal that both suspensions show a substantial weight loss, starting at 80 °C, due to water evaporation. The decrease in the weight stops at approximately 180 °C for both suspensions, indicating additional polycondensation reactions of the melamine-formaldehyde resin in the walls of the microcapsules and, most likely, an evaporation of formaldehyde along with the water. The weight loss at this temperature is much higher for the microcapsules without a core because the suspension with TPP microcapsules is more concentrated than the suspension with blind microcapsules; consequently, more water evaporated from the second sample. TPP microcapsules start to degrade at 320 °C, whereas blind microcapsules start to degrade later, at 360 °C. The decomposition finishes, for both MC types, at approximately 420 °C. A further increase in the temperature does not change the weight of the samples.

The DSC diagram (**Figure 5**) shows an endothermic peak at 150 °C, confirming the evaporation of water and formaldehyde from the suspensions of microcapsules. Blind MCs show high exothermic peaks at (410, 450 and 590) °C, indicating different degrading products of the melamine-resin wall material. TPP microcapsules have a small exothermic peak at 330 °C and no other exothermic peak arises with the increasing temperature. The released heat is, therefore, much lower with TPP than with blind microcapsules. We also assume that the degradation at 330 °C releases the flame retarding products, hindering further burning or oxidation of the surroundings.

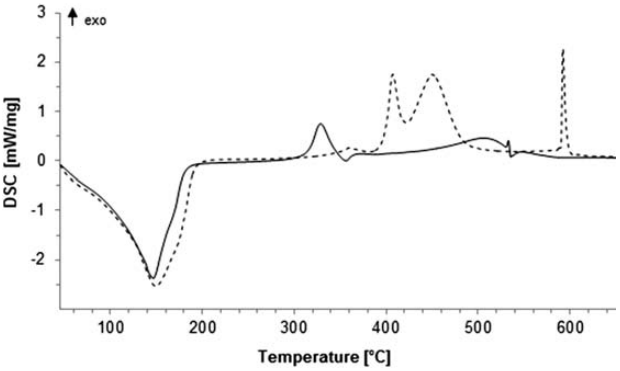

Figure 5: DSC curves of the samples: TPP MCs (——), MCs without a core (- - -)
Slika 5: DSC-krivulje vzorcev: TPP MC (——), MC brez jedra (- - -)

In **Figures 6** to **9**, we can see the TGA and DSC curves of the untreated cotton and polyester and also both samples printed or impregnated with TPP MC. All the diagrams also present a curve of the TPP MC suspension.

At first glance, we can see that the TGA curves for all of the textile samples are very similar. The curve of the suspension deviates from dry samples because of the evaporation of water at the temperatures below 180 °C. All of the CO samples lost their weight at the lower temperatures (330 °C) than the PES samples (400 °C). The printed samples degrade earlier than the raw materials, and the impregnated ones that carry more MCs degrade even earlier. The TPP MCs start to degrade at lower temperatures than the surrounding material, and the degradation products such as P_2, PO, PO_2 and HPO_2 in the vapour phase extinguish the flame. Consequently, if there are more microcapsules on the material, the degradation starts earlier and the flame retardancy is stronger.

The DSC curves in **Figures 7** and **9** confirm that the presence of MCs on the textile materials changes their

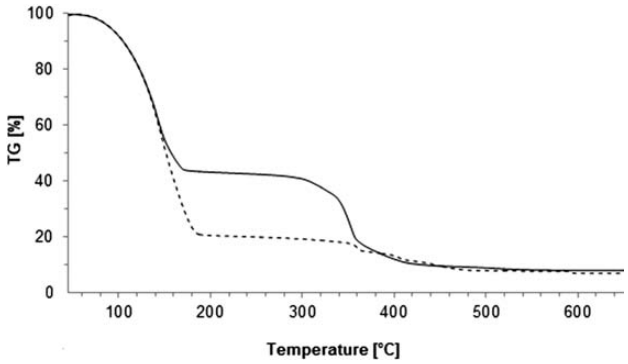

Figure 4: TG curves of the samples: TPP MCs (——), MCs without a core (- - -)
Slika 4: TG-krivulje vzorcev: TPP MC (——), MC brez jedra (- - -)

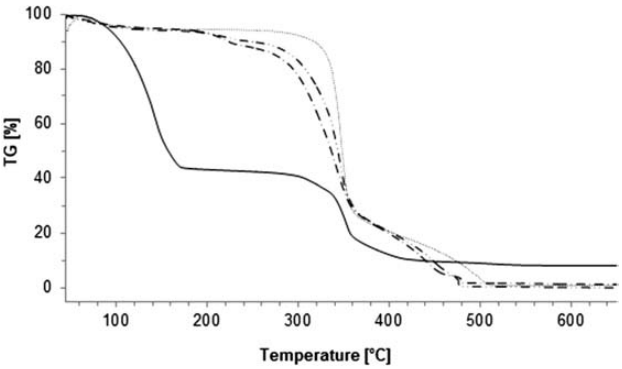

Figure 6: TG curves of the samples: TPP MC (——), CO (·····), COi800 (–·–), COp400 (–··–)
Slika 6: TG-krivulje vzorcev: TPP MC (——), CO (·····), COi800 (–·–), COp400 (–··–)

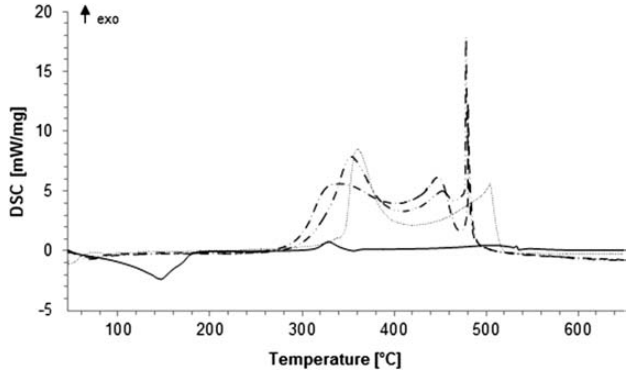

Figure 7: DSC curves of the samples: TPP MC (——), CO (·····), COi800 (–·–), COp400 (–··–)

Slika 7: DSC-krivulje vzorcev: TPP MC (——), CO (·····), COi800 (–·–), COp400 (–··–)

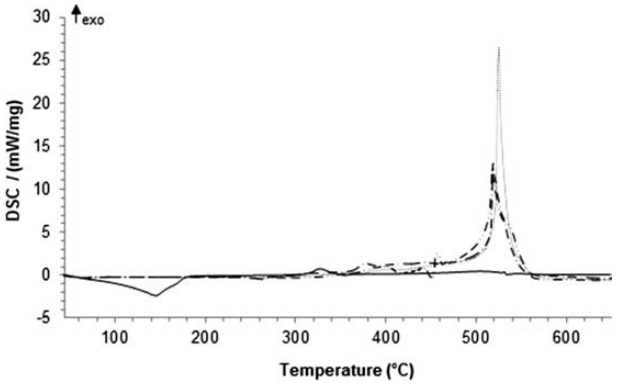

Figure 9: DSC curves of the samples: TPP MC (——), PES (·····), PESi800 (–·–), PESp600 (–··–)

Slika 9: DSC-krivulje vzorcev: TPP MC (——), PES (·····), PESi800 (–·–), PESp600 (–··–)

properties. The printed cotton fabric has lower exothermic peaks, starting at lower temperatures than the raw cotton fabric; even lower are the exothermic peaks of the impregnated samples with more MCs on their surfaces. The microcapsules decrease the heat released from the cotton fabric. A similar picture is seen for the PES fabrics. The exothermic peak is the highest for the untreated PES. The PES samples impregnated or printed with MCs have lower peaks than the untreated sample. Both are very similar. The microcapsules decrease the heat release from the PES fabric as well.

A comparison of **Figures 7** and **9** also demonstrates how different the behaviours of cotton and polyester fibres are at high temperatures. Natural CO fibres degrade gradually in several oxidation reactions, represented by several broad peaks on the DSC curve, whereas synthetic PES fibres degrade almost instantly at higher temperatures. The DSC curve shows one high, sharp peak at approximately 520 °C.

3.4 Fabric properties

The mass per unit areas of raw, printed, impregnated and washed samples are presented in **Figure 10**. The differences in the structure between the woven and nonwoven fabrics influence different absorption and retention parameters of the added substances.

The mass per unit area of the untreated PES samples (184 g/m^2) is higher than that of the untreated CO samples (124 g/m^2). The higher mass and more porous structure of PES enable a greater absorption of the microcapsules from the printing paste and an even higher absorption from the impregnation bath. The highest addition of MCs can be observed on the impregnated PES samples, which also show the best flame retardancy. The compact cotton fabric does not enable a high accumulation of MCs. The mass per unit area increases with the increasing amount of the microcapsules in the impregnating baths and/or printing pastes and it is more pronounced in the PES samples than in the CO samples.

The air permeability (**Figure 11**) of the polyester samples is much higher than that of the cotton samples. The permeability of both evidently decreases with higher amounts of the applied microcapsules. Again, there are larger differences among the polyester samples than among the cotton samples due to higher deposits from the impregnation and/or the printing system on the polyester. For both materials, PES and CO, the impregnated samples have a better air permeability than the printed

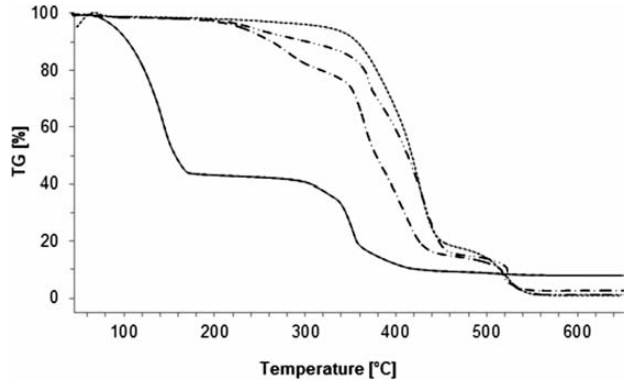

Figure 8: TG curves of the samples: TPP MC (——), PES (·····), PESi800 (–·–), PESp600 (–··–)

Slika 8: TG-krivulje vzorcev: TPP MC (——), PES (·····), PESi800 (–·–), PESp600 (–··–)

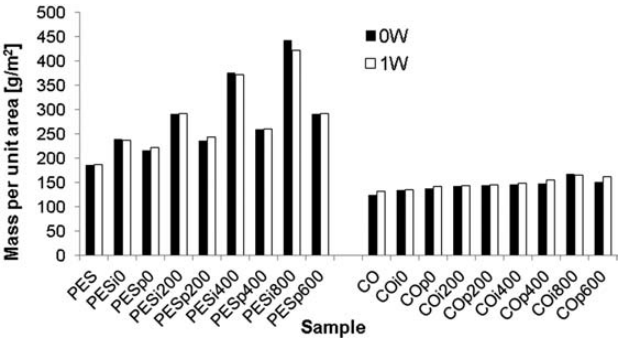

Figure 10: Mass per unit areas of impregnated, printed, unwashed and washed PES and CO samples

Slika 10: Ploščinska masa impregniranih, tiskanih, nepranih in pranih PES- in CO-vzorcev

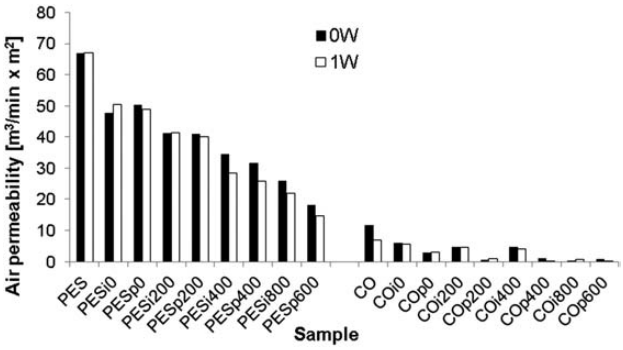

Figure 11: Air permeability of impregnated, printed, unwashed and washed PES and CO samples

Slika 11: Zračna prepustnost impregniranih, tiskanih, nepranih in pranih PES- in CO-vzorcev

ones. Printing on the polymer created a relatively impermeable layer on the samples, while a large quantity of the MCs applied on them led to further impermeability.

All the printed and impregnated samples had a very high rigidity; the rigidity of the impregnated samples was so high that it could not be accurately measured in this study.

4 CONCLUSIONS

Fire retardant triphenyl phosphate was encapsulated via an *in-situ* polymerisation method. The produced microcapsules were round in shape, undamaged and 1–7 μm in size, and they were successfully applied to cotton woven and polyester nonwoven fabrics using screen printing and impregnation techniques.

An addition of TPP microcapsules prolongs the burning time of the PES samples, while it has no influence on the burning time of the CO samples with less than 800 g/kg of the microcapsules. Neither of the materials burned when impregnated with the maximum concentration of microcapsules, 800 g/kg. The LOI of these samples (25) showed that the ignition and burning decreased, but they were not sufficiently disabled for these materials to be considered fire retardant.

The TGA analysis shows that with more microcapsules on the material the degradation starts earlier and the flame retardancy is stronger. The DSC curves reveal that on both PES and CO samples the microcapsules decrease the heat release from the fabric.

Structural differences between the cotton woven fabric and the polyester nonwoven fabric influenced different absorptions of the microcapsules and their contributions. The mass per unit area increased with the increasing amount of the microcapsules in the impregnating baths and/or the printing pastes and was more pronounced in the PES samples than in the CO samples. The impregnated PES samples absorbed the highest amount of MCs and showed the best flame retardancy. The air permeability of both samples decreased with the application of the microcapsules and the rigidity increased drastically.

The results show that the investigated processes are not yet appropriate for practical use and that more research is needed to improve the fire-retardant properties of triphenyl phosphate microcapsules and their influence on the textile properties of the treated fabrics.

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