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# Composite Nonwovens with Natural Additives

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## Abstract

Various methods of nonwoven composite materials manufacturing are known. One such method is the well-known technique called spun-bonding. The production technology for composite nonwoven by the spun-bond method is known, but the technique of introducing an additive in the form of shredded wastes of natural origin so as to obtain a composite nonwoven fabric with interesting functional properties is new. The article describes a method of producing an innovative composite nonwoven using the spun-bond technique. As a result of incorporating various additives into the nonwoven structure, composite nonwovens with modified properties are obtained. Composite nonwovens, depending on the additive used, can be utilised as filtration material in the construction, agriculture or automotive industry.

**Key words:** composites, nonwoven, spun-bond method.

## Introduction

New environmental and economic aspects as well as the depletion of crude oil resources have induced the scientific community to increase interest in the waste recycling problem, including the natural waste of fibrous materials [1-2]. This group includes poultry feathers, cotton fibre wastes, waste paper and dust generated during the milling of MDF boards.

Various methods of nonwoven composite materials manufacturing are known [3]. One such is the well-known technique called spun-bonding. Biodegradable aliphatic polyester nonwoven fabrics, with their excellent heat stability and mechanical strength, are known. The aliphatic polyester nonwoven fabric invented by [4] is useful for sanitary, medical, packaging, and agricultural covering materials, as well as for filters, oil absorption materials and the like.

Composite nonwovens made of bicomponent fibres of the skin-core or side to side type formed from two different polymeric materials and manufactured using the spun-bond method are known [5-11]. Such nonwovens produced have the properties of both polymer components. The process of forming this type of nonwoven involves melting and plasticising both polymer components (intended for the core and skin) by means of separated extruders, then extruding the plasticised polymers through a spinning nozzle of

special design, stretching and solidification of the bicomponent fibres in the air stream, forming a fleece, and then joining it into a nonwoven.

The spun-bond method can also be used to obtain nonwovens from polymer blends from two types of polypropylene characterised by different melting temperatures and melt flow rates [12]. From a mixture of fibre-forming polymer with modifying agents melted together with the polymer, spun-bond nonwoven with increased resistance to ultraviolet radiation is obtained [13].

The production technology of composite nonwoven by the spun-bond method is known, but the method of introducing an additive in the form of shredded wastes of natural origin so as to obtain a composite nonwoven fabric with interesting functional properties is new [14]. The article describes a method of producing an innovative composite nonwoven using the spun-bond technique. As a result of incorporating various additives into the nonwoven structure, composite nonwovens with modified properties are obtained.

The advantage of these nonwovens is the distribution of modifying additives in their whole volume. Composite nonwovens, depending on the additive used, can be utilised as filtration material in the construction, agriculture or automotive industry.

## Objective and scope of work

The aim of the research was to use the spun-bond method for the production of nonwovens made of polypropylene (PP) with the addition of poultry feathers (F)

and other natural additives that are difficult to manage, i.e. dust generated during milling boards (MDF), cotton fibres (CF), and waste paper (WP).

## Materials

The following materials were used for the manufacture of composite nonwovens:

- Polypropylene HP 462R (Basell Orlen Polyolefins Sp. z o.o.), MFI (210 °C) = 25 g/10 min

## Modifying additives (DM)

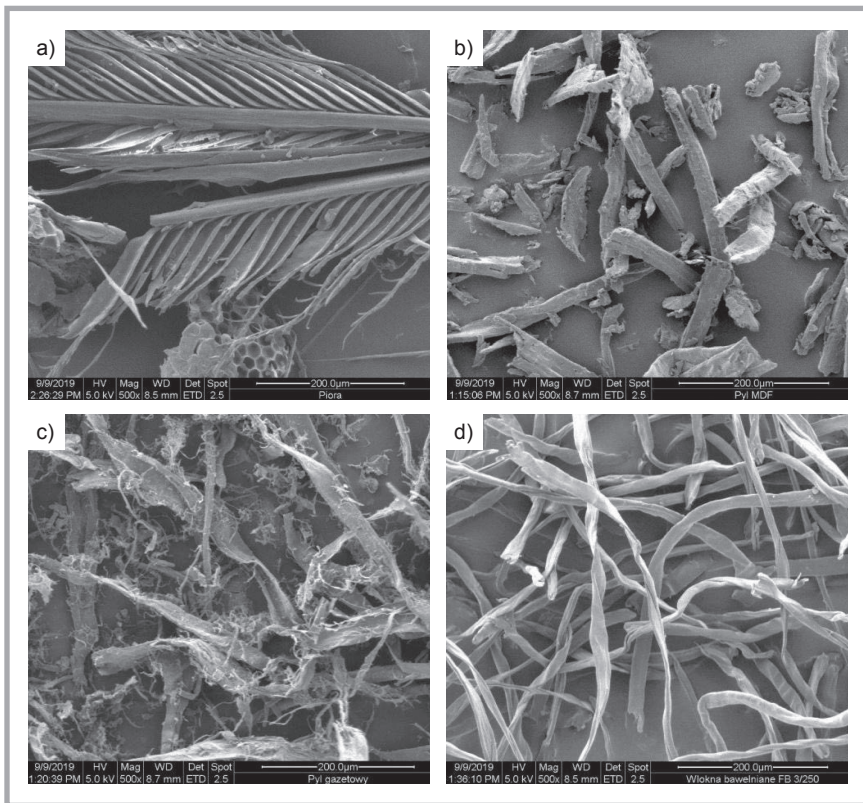
- F – white poultry feathers after physical and mechanical treatment (Poultry Slaughterhouse – Jack. PPHU. Kraska J., Szynekielew)
- MDF – dust generated during milling of MDF boards (IKEA)
- WP – waste paper (so-called newspaper dust)
- CF – cotton fibres FB 3/250 (SCHWARZWALDER TEXTIL – WERKE, Germany)

*Figure 1* presents SEM photos of modifying agents used for PP nonwoven modification.

## Methodology

### Feather pre-treatment

Poultry feather wastes, obtained from a slaughterhouse, were subjected to preliminary pre-treatment consisting of three times washing in hot water with the addition of detergent for 1 hour. After filtering the water, the feathers were treated with 15% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) for 1h and filtered again. The wet feathers were cut into parts of max 6 mm length and then dried.



**Figure 1.** SEM photos : a) F , b) MDF, c) WP and d) CF used for manufacturing composite PP nonwoven by the spun-bond method.

### Manufacture of composite nonwovens with the use of feathers and other additives by the spun-bond method

#### Preparation of a spun-bond forming stand:

A stand for nonwovens formed by the spun-bonded method was equipped with a special dosing system for the introduction of modifying additives into PP. The dosing system (**Figure 2**) for modi-

fiers in the form of dust or fibres consists of the following components:

- charging hopper,
- feeding channel with segmented cylindrical brush and sieve partition,
- inlet channel.

#### Nonwoven forming by the spun-bond method

The technology of producing nonwoven made of filament fibres by the spun-bond method consists in plasticising, melting and extruding the polymer granulate by the extruder, then the polymer alloy flows through the filter to the dosing pump, which transfers the polymer with a constant stream to the multi-hole spinneret head.

In the forming process of composite nonwovens using the spun-bond technique, a directed stream of air containing solid substances in the form of dust, fibres or flakes from natural raw materials is introduced into the cooling and stretching zone of fibres from fibre-forming polymers, which is sucked through the process air.

The polymer streams flowing out of the spinneret head are cooled with a cold stream of air, and then, after solidifica-

tion, the fibres are stretched in a stream of air of increasing speed, which in the final phase undergoes strong turbulence, causing the entanglement of fibres and their chaotic arrangement in the form of a fleece on a movable receiving conveyor. The fleece is welded on hot calender shafts, and the nonwoven fabric obtained is collected on the receiving device.

#### Manufacture of PP nonwoven with the addition of feathers (F)

Polypropylene of melt flow rate MVR = 25 g/10 min was extruded using a single-screw extruder at a temperature of subsequent heating zones of 160 °C, 190 °C and 215 °C, then molten polymer at a temp. of 215 °C and pressure of 1900 hPa was pressed through a spinneret containing 467 capillaries using a pump with a capacity of 0.10 g/min/ capillary. Fibres from the spinneret head were directed to a channel in which the fibres were stretched using cooling air at a temperature of 15 °C and a pressure difference of 1200 hPa. The cooling air sucked the air stream with shredded feathers (**Figure 1.a**) with a particle size from 20 µm to 5 mm. The stretched PP fibres together with the particles of shredded feathers, after passing through the forming channel, fell in the form of a fleece into the transport sieve, under which a pressure 214 Pa lower than atmospheric was created. Then the pre-laminated fleece was calendered at a temperature of 135 °C. The nonwoven was collected on the receiving device at a speed of 2.85 min.

#### Manufacture of PP nonwoven with the addition of MDF dust

Polypropylene of melt flow rate MVR = 25 g/10 min was extruded using a single-screw extruder at a temperature of subsequent heating zones of 160 °C, 190 °C and 215 °C, then molten polymer at a temp. of 215 °C and pressure of 1900 hPa was pressed through a spinneret containing 467 capillaries using a pump with a capacity of 0.10 g/min/capillary. Fibres from the spinneret head were directed to a channel in which the fibres were stretched using cooling air at a temperature of 15 °C and a pressure difference of 1200 hPa. The cooling air sucked the air stream with dust generated from the milling of MDF boards (**Figure 1.b**) with a particle size of no more than 1mm. The stretched PP fibres together with dust particles, after passing through the forming channel, fell in the form of a fleece



**Figure 2.** Fragment of a spun-bond installation with a dosing system: 1 – charging hopper, 2 – feed roller, 3 – inlet channel, 4 – disturbance channel, 5 – transport sieve.

into the transport sieve, under which a pressure 231 Pa lower than atmospheric was created. Then the pre-laminated fleece was calendered at a temperature of 145 °C. The nonwoven was collected on the receiving device at a speed of 2.85 m/min.

#### *Manufacture of PP nonwoven with the addition of waste paper (WP)*

Polypropylene of melt flow rate MVR = 25 g/10 min was extruded using a single-screw extruder at a temperature of subsequent heating zones of 160 °C, 190 °C and 215 °C, then molten polymer at a temp. of 215 °C and pressure of 2020 hPa was pressed through a spinneret containing 467 capillaries using a pump with a capacity of 0.10 g/min/capillary. Fibres from the spinneret head were directed to a channel in which the fibres were stretched using cooling air at a temperature of 15 °C and pressure difference of 1200 hPa. The cooling air sucked the air stream with dust of waste paper (*Figure 1.c*) with a particle size of up to 50 µm. The stretched PP fibres together with newspaper dust, after passing through the forming channel, fell in the form of a fleece into the transport sieve, under which a pressure 187 Pa lower than atmospheric was created. Then the pre-laminated fleece was calendered at a temperature of 135 °C. The nonwoven was collected on the receiving device at a speed of 2.85 m/min.

#### *Manufacture of PP nonwoven with the addition of cotton fibres (CF)*

Polypropylene of melt flow rate MVR = 25 g/10 min was extruded using a single-screw extruder at a temperature of subsequent heating zones of 160 °C, 190 °C and 215 °C, then molten polymer at a temp. of 215 °C and pressure of 1240 hPa was pressed through a spinneret containing 467 capillaries using a pump with a capacity of 0.07 g/min/capillary. Fibres from the spinneret head were directed to a channel in which the fibres were cooled and stretched. The cooling air sucked the air stream with cotton fibres FB 3/250 (*Figure 1.d*) of 1.8 mm length, 3-40 µm thickness, 35 g/l bulk density and 2.8% humidity. The stretched PP fibres together with cotton fibres, after passing through the forming channel, fell in the form of a fleece into the transport sieve, under which a pressure 189 Pa lower than atmospheric was created. Then the pre-laminated fleece was calendered at a temperature

of 135 °C. The nonwoven was collected on the receiving device at a speed of 1.9 m/min.

## ■ Analytical methods

### Assessment of mechanical parameters of nonwovens

Assessment of the mechanical properties of the composite nonwovens was carried out in the Laboratory of the Experimental and Production Plant of IBWCh according to the following standards:

- Basis weight according to PN-ISO 536: 1996
- Determination of tensile strength according to the standard: PN-EN ISO 1924-2: 2009

The test was carried out according to the following:

Tensile strength: initial length of the specimen 200 mm, rate of extension  $V = 100$  mm/min, width of the specimen 50 mm, number of test specimens  $n=5$ .

### Determination of pore size by the bubble method

Determination of the pore size was carried out according to the procedure described in the standards of the American Society for Testing and Materials, method F316.

The bubble point method is the most widely used for pore size determination. It is based on the fact that for a given fluid and pore size with a constant wetting, the pressure required to force an air bubble through the pore is inversely proportional to the size of the hole.

In practice, the pore size of the filter element can be established by wetting the element with fluid and measuring the pressure at which the first stream of bubbles is emitted from the upper surface of the element.

The theoretical relation between this transition pressure and bubble-point pressure is:

$$D = (4g \times \cos q)/P$$

where:

- P – bubble-point pressure,
- g – surface tension of the liquid (72 dynes/cm for water),
- q – liquid-solid contact angle (which for water is generally assumed to be zero),
- D – diameter of the pore.

### Air permeability

Testing of the air permeability was carried out according to ISO 9237 “Determination of air permeability of textiles”. The measurement method consists of passing air through the sample at a pressure difference of 2 mbar. The measure of permeability is the velocity of air flow through the sample tested, expressed in  $l/m^2s$ .

### Study of the structure of composite nonwovens using scanning electron microscopy (SEM)

Structural tests of the external surface and cross-section of the composite nonwovens were carried out using a scanning microscope – Quanta 200 (FEI Co., USA), under a high vacuum at an electron accelerating voltage of 2KV. Before being placed into the microscope chamber, samples were covered with a thin layer of gold by means of a vacuum sprayer – Q150R S.

### Thermal analysis

Thermal analysis was performed using a TG Labsys Evo 1150 (Setaram) instrument with control software, and the results were processed by “Calisto” AKTS. The tests were performed in an anaerobic nitrogen atmosphere at a flow of 21  $cm^3/min$ , using TG-DSC analysis. Samples with a mass of 12.8 ÷ 17.3 mg placed in Pt 100 µl cells were heated at a rate of 5 °C/min in the temperature range of 20 ÷ 600 °C.

## ■ Results and discussion

As a result of introducing various additives into the nonwoven structure, composite nonwovens with modified properties are obtained.

### Assessment of thermal properties of modifying additives (DM) and composite nonwovens PP/DM

The study of thermal stability using TGA and DSC analyses is the basic to characterise materials in polypropylene (PP) processing.

The introduction of additional components to PP involves the need to assess their impact on the PP degradation process. The purpose of thermal analysis is therefore to determine how the components used in the study affect the TGA and DSC thermograms of PP/DM nonwoven composites. Shown below are selected

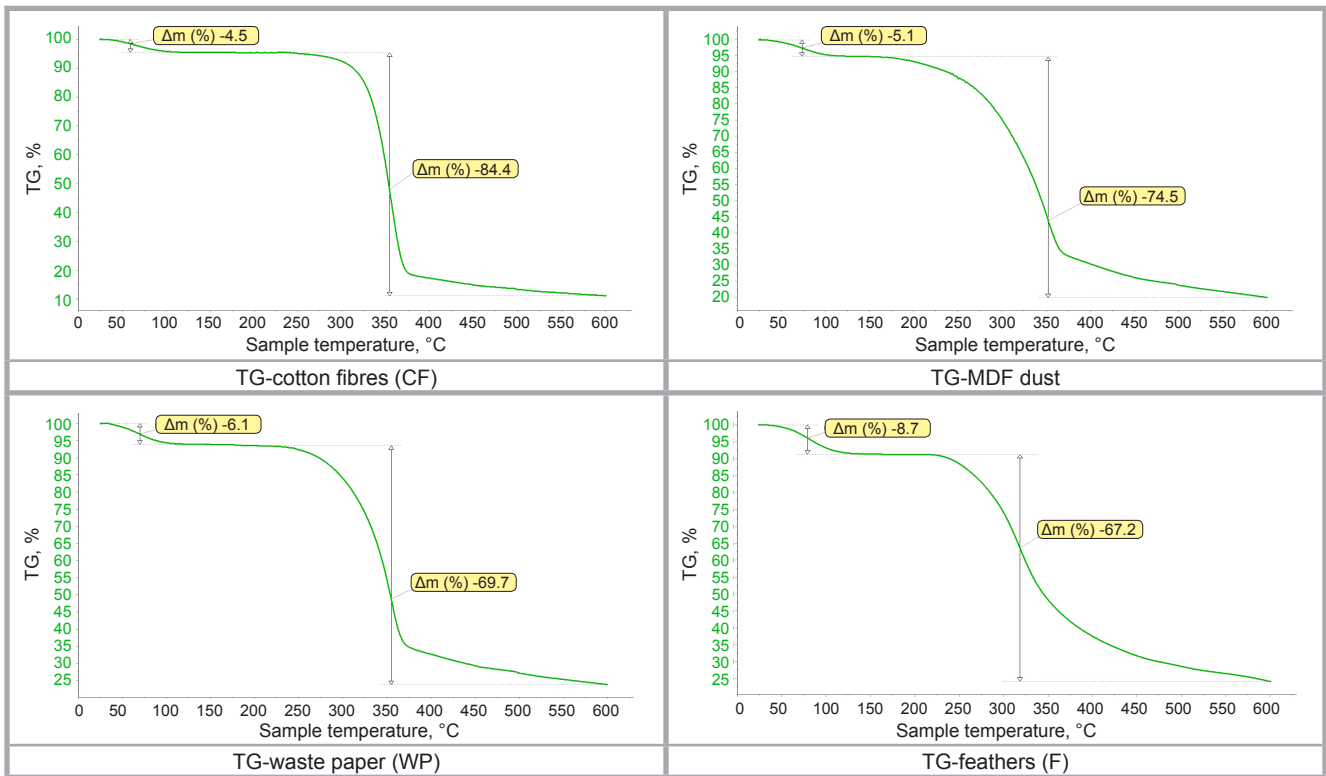


Figure 3. Thermal degradation of additives used for PP nonwoven modification: TG curves: 1) CF; 2) MDF; 3) WP; 4) F.

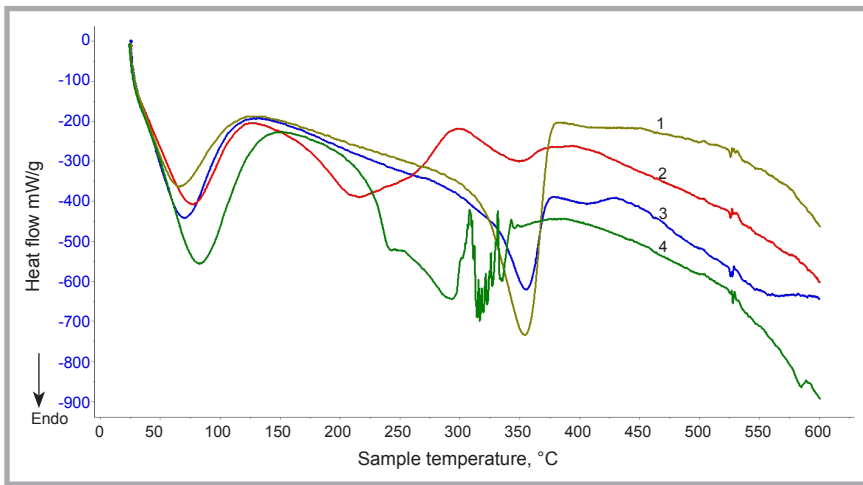


Figure 4. Thermal degradation of additives used for PP nonwoven modification: DSC curves: 1) CF; 2) MDF; 3) WP; 4) F.

thermograms of the additives introduced into the PP nonwoven (Figures 3 and 4) and for nonwoven composites with additives PP/DM (Figure 5) compared to the PP nonwoven.

**Thermal analysis of modifying additives**

Thermogravimetric analysis indicates a similar character of the phenomena in all samples tested. The results of thermal tests of additives in the form of cotton fibre, MDF dust, newspaper dust and poultry feathers obtained indicate a two-stage degradation process in each sample

tested. The first stage, mainly related to water desorption, occurs up to approx. 114 °C (4.5-8.7%). During further heating, the largest mass loss related to thermal decomposition is observed for cotton fibres and the smallest one for feathers (Table 1).

DSC curves (Figure 4) have the character of an endothermic process. Clear peaks in the range of lower temperatures up to about 150 °C are mainly associated with water evaporation. Their size correlates with the percentage weight loss related to, among others, water evapo-

ration (Table 1). Further peaks appear in the range of higher temperatures (above 150 °C) and are characteristic for a given type of additive. For newspaper dust and cotton fibres, there are individual peaks at a temp. of 355 °C and 354 °C, respectively, which are associated with thermal degradation of the cellulosic component.

For MDF dust two peaks in the higher temperature range are observed, the first at 216 °C, for which the binder used for bonding MDF may be responsible, the second at 350 °C, associated mainly with thermal degradation processes.

Poultry feathers are characterised by a complex decomposition process associated with protein degradation, which begins above 160 °C. Using the TG and DSC methods, changes in the structure of the modifying additives were analysed to enable detailed analysis of composite nonwovens produced containing them.

**Thermal analysis of PP nonwovens with additives**

Samples of PP nonwovens with the addition of cotton fibres, MDF dust and newspaper dust and PP nonwoven without additives (as a reference sample) were subjected to testing. The test was carried out in the temperature range 20-600 °C. The above-mentioned additives used in

composites degrade at a lower temperature than the polymer matrix, and they determine the total thermal resistance of the composite (Table 2). However, thermal parameters remain at a level that allows to process and obtain functional composites.

The polymer matrix decomposes almost 100% (residue heating – heating to 600 °C; 0.5%), while decomposition of the composite at a temperature of 600 °C leaves products of additive degradation (Table 2). For the PP reference nonwoven, a weight loss of 1.5% is noticeable in the melting area, which may be related to the release of low molecular weight substances. However, in the case of composites, the range of DSC and TG curves for melting PP nonwoven is more complicated because the effects of evaporation of water from the additives as well as those caused by chemo-physical changes in various impurities present in these additives are observed.

Analysis of TG and DSC curves (Figure 5) shows that the degradation process of nonwovens with additives proceeds in two stages. The first stage of composite degradation (Table 2) is the effect of the degradation of the additive used in the composite (cotton, MDF dust, newspaper dust), while the second one is the thermal degradation of the polymer matrix. In the case of the initial PP nonwoven (reference), a one-stage effect of weight loss is observed (Table 2). On the DSC curves, endothermic peaks for all samples tested are visible in the temperature range 165.5-169.8 °C, the peak relates to the melting of PP in all four samples. At a temperature above 170 °C, there are peaks corresponding to the degradation of the polymer, as well as the polymer and additive in the case of composites (Figure 5). For the nonwoven/MDF sample the temperatures are 361.6 °C and 458.3 °C, for the non-woven/ cotton fibres – 370 °C and 451.3 °C, for the nonwoven/newspaper dust – 454.6 °C. and for the PP reference non-woven – 445.7 °C.

### Assessment of mechanical properties of PP/DM composite nonwoven

For the nonwovens obtained, the mechanical properties were determined (Table 3). The amount of additive introduced was determined indirectly as the difference between the basis weight of the nonwoven with the additive and that of the initial nonwoven (reference) formed under the same conditions.

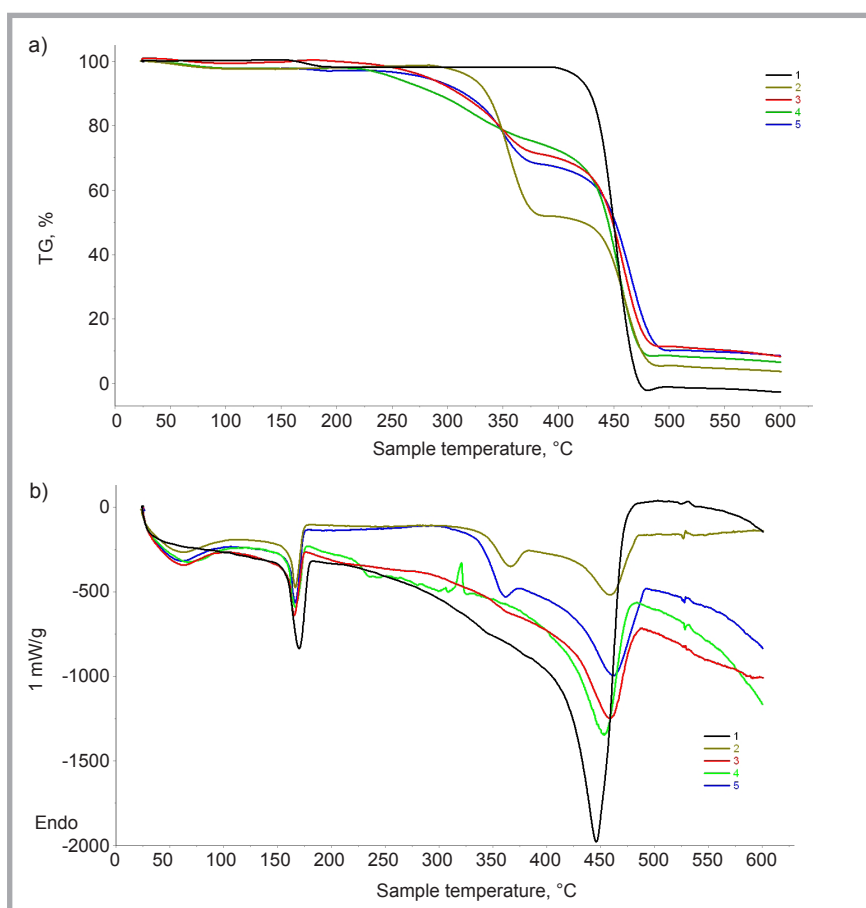


Figure 5. Thermal degradation of composite nonwovens: a) TG Thermogram and b) DSC Thermogram: 1 – PP nonwoven, 2 – PP/CF nonwoven, 3 – PP/ MDF nonwoven, 4 – PP/F nonwoven, 5 – PP/WP nonwoven.

Table 1. Characteristics of the transformation occurring during the heating of additives used for PP nonwoven modification. Note:  $\Delta m$  – loss of weight,  $T_1$  – temperature of the effective start of the weight loss process determined according to the company's program using the horizontal method,  $T_{inf}$  – maximum temperature of the mass loss process,  $T_2$  – temperature of the effective end of the weight loss process determined according to the company's program using the horizontal method,  $\Delta m$  – were determined in relation to the mass of the initial sample.

Sample	Water evaporation				Thermal decomposition				Residue after measurement at 600 °C
	$\Delta m$ , %	$T_1$ , °C	$T_{inf}$ , °C	$T_2$ , °C	$T_1$ , °C	$T_{inf}$ , °C	$T_2$ , °C	$\Delta m$ , %	$\Delta m$ , %
CF	4.5	42.2	59.3	91.7	339.7	354.0	365.1	84.4	11.0
MDF	5.1	49.8	73.2	97.5	314.3	352.3	370.0	74.5	20.1
WP	6.1	47.3	69.9	91.6	319.4	354.8	374.7	69.7	23.8
F	8.7	51.4	79.2	113.4	277.6	318.3	376.1	67.2	24.1

Table 2. Thermal characteristics of changes occurring during the heating of PP nonwoven and PP/DM composite nonwovens. Note:  $\Delta m$  – loss of weight,  $T_1$  – temperature of the effective start of the weight loss process determined according to the company's program using the horizontal method,  $T_{inf}$  – maximum temperature of the mass loss process,  $T_2$  – temperature of the effective end of the weight loss process determined according to the company's program using the horizontal method,  $\Delta m$  – were determined in relation to the mass of the initial sample.

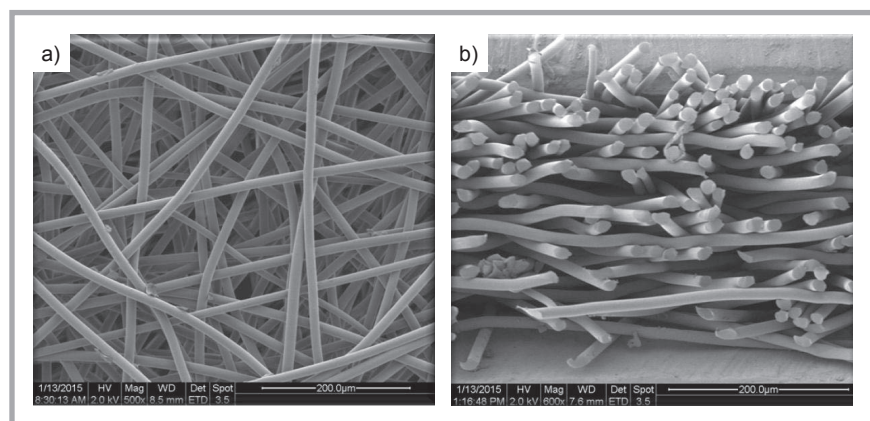
Nonwoven sample	Thermal decomposition								Residue after measurement at 600 °C
	1 stage				2 stage				$\Delta m$ , %
	$\Delta m$ , %	$T_1$ , °C	$T_{inf}$ , °C	$T_2$ , °C	$T_1$ , °C	$T_{inf}$ , °C	$T_2$ , °C	$\Delta m$ , %	
PP/CF	47.1	325.9	355.4	377.6	441.6	460.4	475.6	46.6	3.7
PP/ MDF	29.5	286.3	349.2	374.9	433.3	457.5	480.3	61.7	8.4
PP/WP	29.6	310.6	350.2	372.0	440.0	465.8	483.6	57.2	8.6
PP/F	27.3	248.3	319.3	395.2	430.7	454.2	472.7	62.4	6.6
PP	98.0	438.4	451.9	463.4	–	–	–	–	0.5

**Table 3.** Mechanical properties of PP/DM nonwovens.

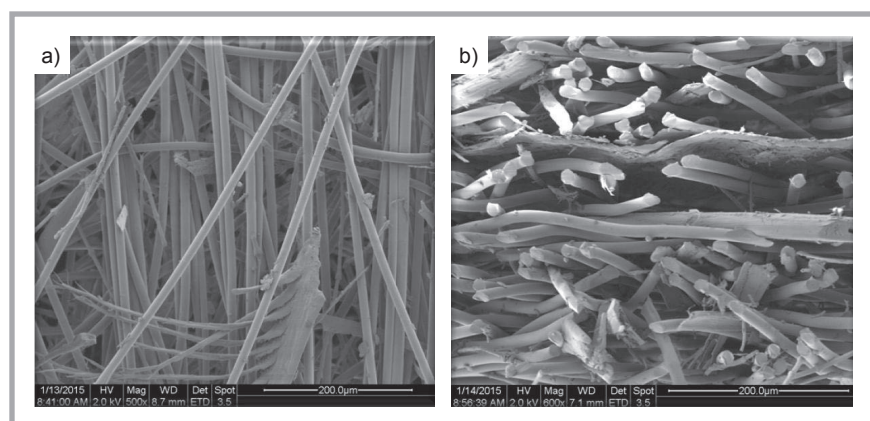
Modifying additive DM	Amount of additive %	Breaking force N	Elongation at break %
0	–	33.9	17.2
CF	20.7	20.7	23.9
MDF	67.2	17.1	9.8
WP	24.9	17.2	27.4
F	37.2	15.4	26.2

**Table 4.** Filtration properties of PP /DM nonwovens. *Note:* \* ratio of average pore size to pore max size.

Sample	Air permeability	Max pores	Average pores	Homogeneity factor*
	l/ms <sup>2</sup>	µm	µm	–
PP	904	248.6	60.6	0.24
F	530	646.4	161.6	0.25
CF	615	142.6	45.7	0.32
WP	905	89.8	60.6	0.67
MDF	209	372.9	47.1	0.13



**Figure 6.** SEM photos: a) PP nonwoven surface, b) PP nonwoven cross-section.



**Figure 7.** SEM photos: a) PP/F nonwoven surface, b) PP/F nonwoven cross-section.

On the basis of the research, it was found that the spun-bond method can be used for the manufacture of composite nonwovens with varying amounts of natural additives which do not meet the criterion required in the spun-bond forming method, i.e. they are not a thermoplastic polymer with a determined MFI value at the temperature of processing. Based

on the results obtained, it is clear that all additives reduce the strength parameters of nonwovens. Poultry feathers, cotton fibres and newspaper dust cause an increase in elongation at break relative to the initial PP nonwoven.

For fibrous materials, a decrease in the breaking force is often observed while

the elongation at break increases, which is advantageous for specific applications, e.g. as a nonwoven base of cosmetic masks.

#### Assessment of filtration properties of PP/DM nonwovens

The results obtained (Table 4) prove that the addition of paper dust (WP) and cotton fibres (CF) to filter materials is suitable. The addition of WP results in obtaining non-woven with a more homogeneous structure, maintaining air permeability at the same level in relation to the PP non-woven fabric, which brings about the improvement of filtration properties. Cotton fibres (CF) cause a decrease in air permeability but at the same time improve the filtration efficiency and homogeneity of the nonwoven, which leads to an improvement in filtration properties.

It was found that poultry feathers (F) and MDF dust are not suitable as an additive to filter materials. Feather addition causes a decrease in air permeability by more than 40%, with a simultaneous decrease in filtration efficiency (max and average pores over 2.5 times larger compared to PP nonwoven), which results in a deterioration of filtration properties. For the PP/MDF composite a clear decrease in air permeability was noted (over 4 times) with a simultaneous deterioration of the homogeneity of the composite nonwoven (homogeneity factor – 0.13) in comparison to the PP nonwoven (Table 4).

#### Assessment of structural properties of composite PP/DM nonwovens using scanning electron microscopy SEM

SEM photos of the surface and cross-section of the PP nonwoven and PP composite nonwovens with various additives are presented below (Figures 6-10).

In the SEM pictures of PP/DM composite nonwovens, clearly seen are the “torn” natural fibres of the additives incorporated, which are enclosed in the structure of the final product. The fibre system creates a characteristically unique surface pattern of the hybrid composite. The image shows both synthetic and natural fibres.

Presented below is a photo of PP composite nonwovens with the addition of ground poultry feathers (F), newspaper dust (WP), dust from milling boards (MDF), and cotton fibre waste (CF) (Figure 11).

## Summary

The advantage of composite spun-bonded nonwovens is the distribution of modifying additives in the whole of their volume. Composite nonwovens, depending on the additive used, can be used as filtration material in the construction, agricultural or automotive industries. Nonwovens manufactured with the addition of waste materials are a new assortment of nonwoven-based products thanks to which wastes of natural origin, difficult to manage, are not stored in landfills.

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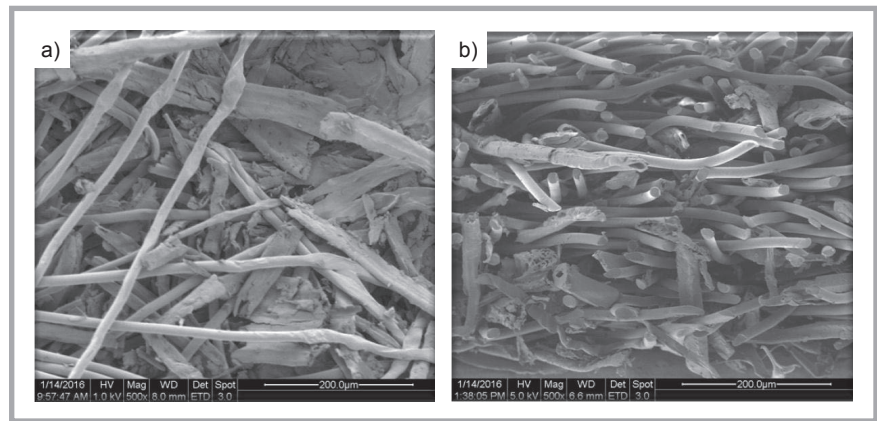


Figure 8. SEM photos: a) PP/MDF nonwoven surface, b) PP/MDF nonwoven cross-section.

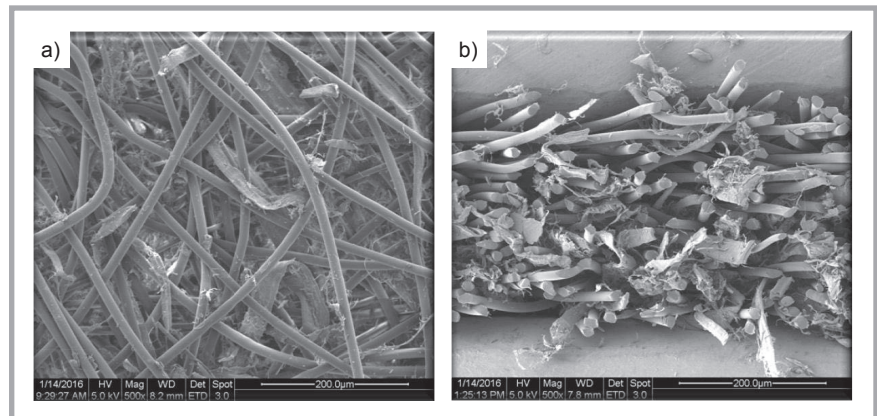


Figure 9. SEM photos: a) PP/WP nonwoven surface, b) PP/WP nonwoven cross-section.

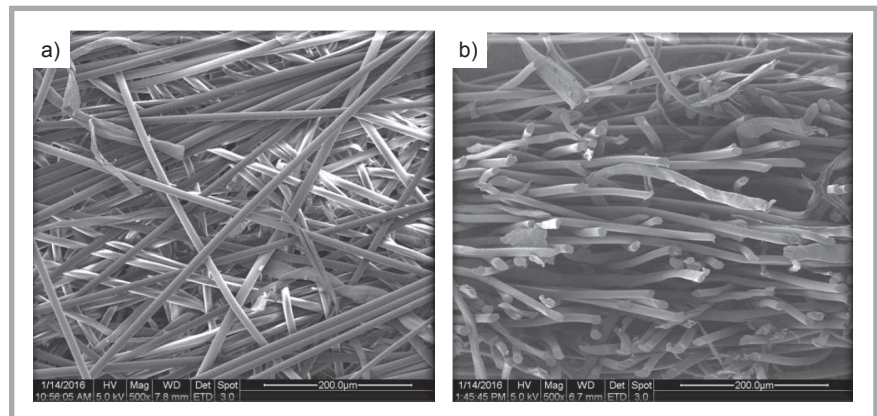


Figure 10. SEM photos: a) PP/CF nonwoven surface, b) PP/CF nonwoven cross-section.



Figure 11. Photo of PP/DM composite nonwovens (from the left: PP/F, PP/WP, PP/MDF, PP/CF).