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CHARACTERISTICS OF FIBRES BASED ON SECONDARY RAW MATERIALS AND THEIR USE IN CONCRETE TECHNOLOGY

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Abstract. Different types of fibres in cementitious composites, particularly in concrete, are currently used for a number of reasons. Fibres are being added to improve mechanical properties (especially steel and glass fibres), to increase the durability – to reduce occurrence of microcracks during the concrete aging (mainly synthetic and cellulose fibres), or to increase the fire resistance (polypropylene fibres). Within the study, characterization of different types of alternative fibres (fibres generated during waste recycling that would otherwise end in incinerators or landfills) with possible use in cementitious composites. These were fibres from recycled PET bottles, paper, and mineral wool, whose properties were compared to the traditionally used cellulose and polypropylene fibres. In the experimental part, the thickness, length, shape, and surface of individual fibres were monitored by an optical microscope. Furthermore, the amount of heat of combustion was determined by the calorimetric method, and the differential thermal analysis (DTA) was carried out for determination of the impact of high temperature on monitored fibres. The microstructure of fibres was monitored using a scanning electron microscope. The focus of the experimental study was on fibres usable in concrete and capable of enduring high temperature stress.

Keywords: Fibre reinforcement, waste fibres, temperature resistance.

1. Introduction

The ecological impact of individual industrial fields belongs to the key issues of the present time. Effort is being made, using constantly evolving modern technologies, to make more use of secondary or alternative raw materials as a substitute for primary raw materials and, at the same time, implement new recycling technologies that will allow transform the waste into the secondary raw materials for other possible industrial usage, including construction. In the given case, these are also renewable raw materials belonging mainly to natural organic raw materials that can be used together with the secondary raw materials in the fibre form even within the concrete technology, and so improve the resulting properties of the composite and possibly influence its behaviour under conditions of extreme stress (e.g., in the case of fire resistance).

Current requirements for fibres added to concrete are regulated by the EN 14889 standards, where the specific requirements for fibres are set regarding their material characteristics. The standard divides the requirements separately for steel and polymer fibres. The requirements for steel fibres are regulated by the EN 14889-1 [1] Fibres for concrete – Part 1: Steel fibres. The use of polymer fibres is regulated by the EN 14889-2 Fibres for concrete – Part 2: Polymer fibres. Other fibre types are considered suitable providing they meet the requirements of issued technical standards for which a building technical certificate is issued in the Czech Republic. Since the full effectiveness of the EN 14889-2 [2] standard, i.e., since 2006, it is allowed to sell only fibres for concrete marked as CE [3] within the whole European Union.

Fibres for concrete are an important ingredient that allows the modification of concrete properties to increase its useful properties based on applying the benefits of a particular type of fibre. Especially increasing the tensile, form, and bending strength of the material. At the same time, some kinds of fibres, both natural and synthetic, positively influence concrete behaviour in terms of volume changes of the material, namely during shrinking [4–6]. Polypropylene fibres prevent the formation of cracks during the concrete shrinkage [7], both in the solidification stage and also in the hardened concrete. The ability to increase the specific properties of concrete depends especially on the material from which the fibres are made of. Fibres are also an important part of concrete where increased resistance to high temperatures is required (fire resistance).

The increase in concrete durability at high temperatures using fibres is provided by elimination of so-called explosive spalling. Based on several studies [8–11], it has been proven that explosive spalling is caused by increasing pressure of water vapours in a construction, which gradually pass from the heated surface towards a cooler part of the concrete. When the dew point is reached, water vapor condensates into water that gradually fills the porous structure and prevents further passage of water vapor that increases the internal stress of concrete. When internal stress exceeds the value of the tensile strength of concrete, an explosive cracking occurs. Characteristics of the optimal size of fibres for increasing the resistance against explosive spalling were solved within numerous papers [12–14]. When using any polypropylene microfibres in concrete, the spalling is reduced. However, with decreasing fibre cross-sectional size (below 32 µm) and length (6–12), more positive results in terms of higher resistance to the explosive spalling are obtained. Fibres shorter than 6 mm and longer than 12 mm are less effective regarding explosive spalling [12].

As part of the study, the basic characteristics of fibres already used in concrete (polypropylene fibres – PP, cellulose fibres – CEL I) were compared to 3 types of fibres produced by recycling of waste or secondary raw materials (cellulose fibres from recycled paper – CEL II, fibres from recycled PET bottles – PET, and fibres from recycled waste mineral wool – MW). A study of microstructure using an optical and scanning electron microscope was carried out for those 5 types of fibres, together with a behavioural study of these fibres at higher temperatures using a thermal analysis and determination of the heat of combustion. The study was carried out to assess possibilities of using alternative types of fibres for concrete with a capability to withstand exposure to high temperatures.

2. Types of fibres used

As part of the experimental part of the study, characterisation of five types of fibres was carried out. The first two types were fibres which are already industrially produced and used for concrete (PP, CEL I). The other three types were developed fibres that are being produced by recycling old materials whose service live has ended and would end in incinerators or landfills (CEL II, PET, MW). Namely, it was these types of fibres:

- polypropylene fibres – designation PP,
- cellulose fibres – CEL I and CEL II designations (2 types),
- polyethylene terephthalate fibres – PET,
- Fibres from recycled mineral wool – MW.

2.1. Polypropylene fibres – PP

Polypropylene fibres have a wide scale of usage (sprayed concrete, floors, prefabrication) thanks to their specific properties. Related to this are several possible methods of production and geometric properties, which are set out in the standard EN 14889-2 for polymer fibres. These fibres are used to reduce the occurrence of shrinkage cracks; they decelerate the concrete sweating, reduce permeability, and prevent ripping out concrete when removing the formwork. Hardened concrete with these fibres is more resistant against abrasion, impacts, and aggressive cycles [15, 16].

2.2. Cellulose fibres

Cellulose fibres have similar usage as the polypropylene ones (sprayed concrete, prefabrication, strength). It is possible to produce cellulose fibres by two methods. The first method is from natural sources (wood, plants, cotton), where special treatments are used to gain cellulose that is subsequently transformed into fibres. The second method for obtaining cellulose fibres is by a chemical or mechanical treatment of old paper and subsequent garneting. Cellulose fibres are hygroscopic and are able to absorb humidity up to 85 % of their weight and keep it around a fibre. This results in good anchoring and bonding between the fibres and the matrix. Contrary to glass fibres, they do not corrode in the highly acidic environment of concrete. The use of cellulose fibres in concrete increases the resistance to impact, resistance against shrinkage cracks, resistance against freezing cycles, and more [17, 18].

2.3. Polyethylene terephthalate fibres – PET

This type of fibres is produced by recycling PET bottles that cannot be used within the original purpose. Fibres can be produced in several ways. The most used methods are mechanical separation using cutting tools or melting of recycled PET bottles into a melt, creating an infinite fibre, which is subsequently reduced to a desired length. By using recycled PET fibres for concrete, it is possible to obtain concrete of higher stiffness and impact resistance since occurring cracks do not spread freely through the construction element and the stress is distributed to a larger surface by a fibre web [19].

2.4. Fibres from recycled mineral wool – MW

Fibres from recycled mineral wool are being developed in order to process the leftover waste wool from construction sites and their subsequent use. Mineral wool is mechanically separated into smaller parts which are mechanically spread into individual fibres. Fibres for concrete will be added to reduce the occurrence of cracks during concrete aging in the early stages, and thus to increase the durability of the concrete.

Fibres, on which the experimental study was executed, can be seen in Figure 1 below, whereby cellulose fibres (CEL I, CEL II) for both variants were processed into pellets that should reduce dustiness and their garneting occurs during their processing in the cementitious composite.

3. Methodology and measurement results

The determination of important properties for further use of these fibres in concrete demanding to high temperature stress was carried out in prepared fibre samples. These tests were carried out pursuant to the relevant testing standards:

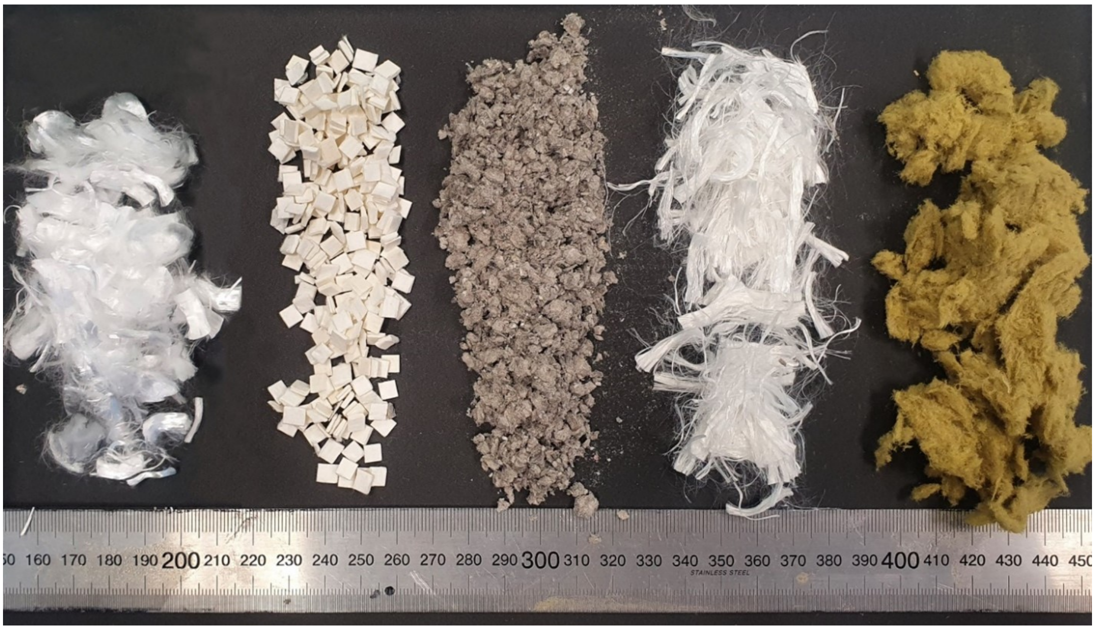

Figure 1. Types of fibres used from the left – PP, CEL I, CEL II, PET, MW.

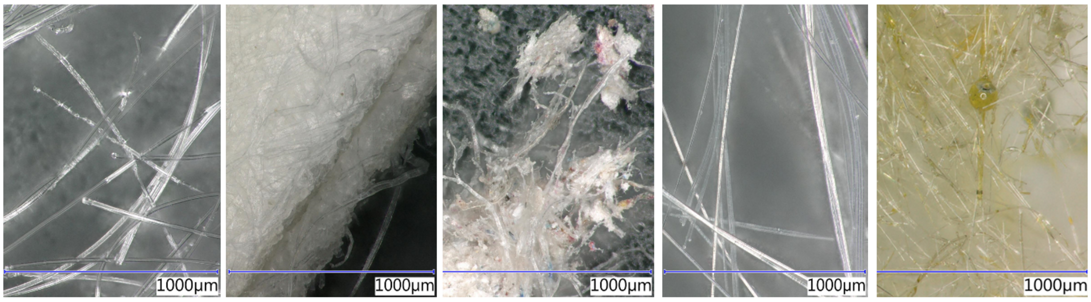

Figure 2. Observed fibres under an optical microscope – PP, CEL I, CEL II, PET, MW.

Fibre type / property	**PP**	**CEL I**	**CEL II**	**PET**	**MW**
Thickness [µm]	20–23	24–36	10–22	13–15	8–15
Face	straight	wavy	variant	straight	straight
Surface	smooth	smooth	rough	smooth	smooth
Length [mm]*	12.12	2.53	0.96	5.98	1.97

*the length of fibres was set to on at least 20 randomly chosen fibres from which the average length was derived.

Table 1. Evaluation of observed fibres under an optical microscope.

- determination of basic properties (length and thickness) under an optical microscope,
- thermogravimetric analysis – TGA, to verify the weight loss of fibres during heating,
- determination of heat of combustion (EN ISO 1716),
- study of the microstructure using a scanning electron microscope (SEM).

3.1. Determination of properties on an optical microscope

The thickness, length and shape of chosen fibre types were determined by an optical microscope at various magnifications. The record was taken using a digital optical microscope Keyence VHX. Images from the optical microscope are shown in Figure 2 at a magnification of 65. Monitored properties are stated in Table 1.

3.2. Thermogravimetric Analysis (TGA)

Within the monitoring of increasing/decreasing weight of chosen types of fibres at increasing temperature, the fibres undergone thermogravimetric analysis (TGA). This method is based on the principle of heating a certain amount of material in which the increase or decrease of its mass is monitored subsequently. In terms of monitored fibres, this analysis is important for the determination of a temperature interval when the onset of melting and subsequent burning (evaporation) of the fibres occurs. Results are shown in the following

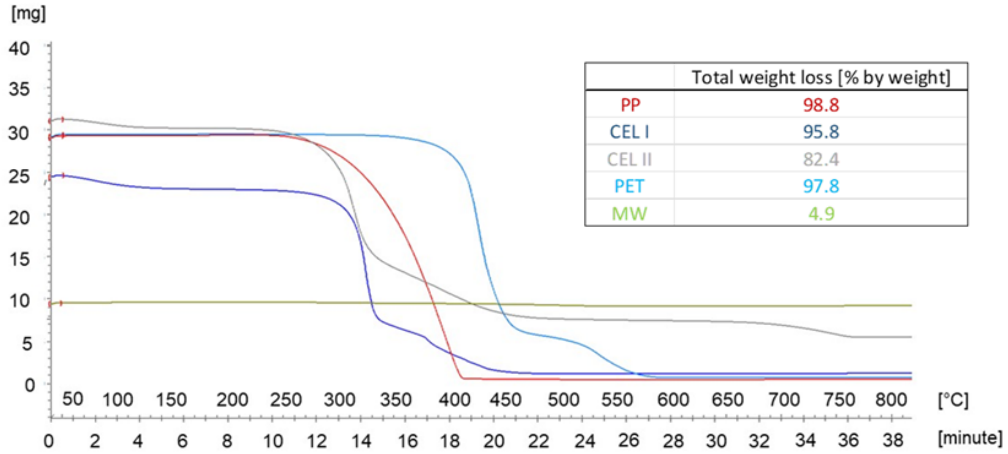

Figure 3. TGA recording of observed fibres.

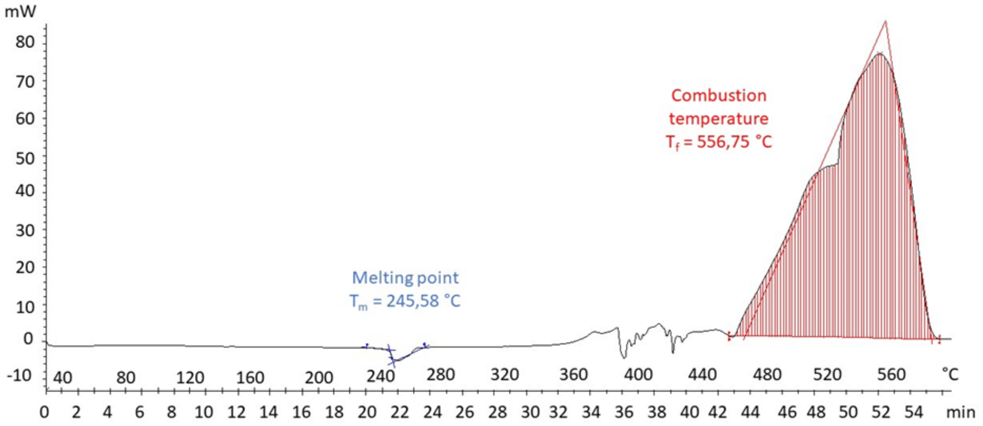

Figure 4. DSC recording from PET fibres.

Figure 3. The X-axis is showing the temperature [°C] while Y-axis is showing the sample weight [mg].

The fastest mass loss occurs for PP fibres in the range of 250–420 °C and, at a temperature of approximately 470 °C, complete degradation of the fibre is completed (98.8 % of mass was decomposed). For cellulose fibres CEL I and CEL II, similar progress can be seen in the graphic evaluation. Approximately around 250–270 °C (CEL II), 290–300 °C (CEL I), there is a slight mass loss due to the release of free and physically bonded water; upon further increasing of temperature, there is a rapid mass loss as a result of the degradation of the cellulose fibre. In the case of recycled cellulose fibres (CEL II), a residual mass of about 18 % of the original mass remains even after reaching 750 °C. In the case of PET fibres, the significant mass loss occurs within the range of 350-580 °C. After reaching 580 °C, a complete degradation of PET fibres occurred (mass balance after test: 2 %). For fibre samples from recycled mineral wool (MW), there was only a minimal fluctuation in mass, which eventually dropped only by 5 %. This behaviour was expected due to the differences in material characteristics and primary raw material used for this type of a fibre (particularly its higher melting point).

3.3. Differential scanning calorimetry (DSC)

From the point of view of determining the effect of high temperature on the heat development during heating, the DSC analysis was carried out on selected types of fibres. This method is based on a constant heating rate of two vessels, one of which is empty, and the second one is carrying the sample. A control unit ensures the constant heating rate of 10 °C/min (within the temperature range of 100–600 °C). The difference in heat fluxes between both vessels is measured. The DSC analysis was carried out on all types of monitored fibres. In terms of applicability in the field of increasing the fire resistance of cementitious composites, the focus was on two polymer fibres – namely PET and PP fibres. The DSC analysis of both fibre types can be seen in Figures 4 and 5, where the melting temperatures of both types is clearly stated, which is very important because volume changes of the fibre occur upon its liquefaction, thus preventing explosive spalling of the cementitious composite.

The melting temperature (point) Tm and the combustion temperature Tf for PET and PP fibres were recorded using the DSC analysis. The melting temperature for PP is approximately 74 °C lower than

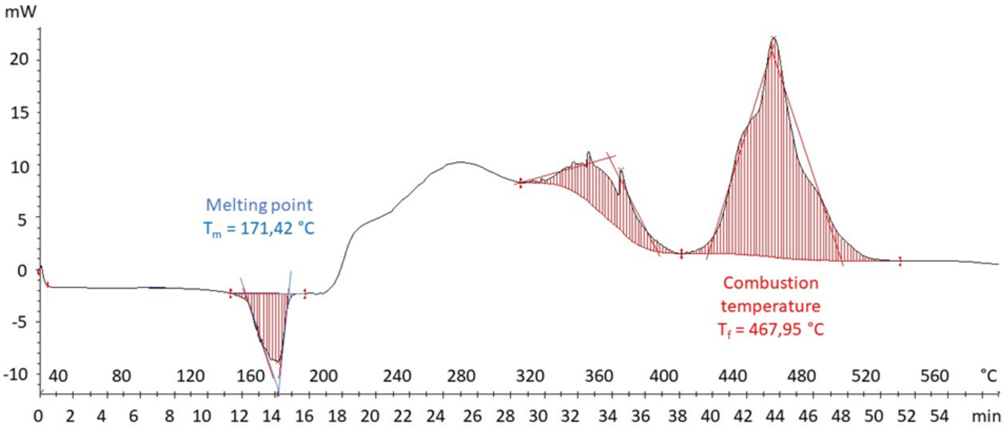

Figure 5. DSC recording from PP fibres.

that of PET; combustion occurred for both types at temperatures above 500 °C. Cellulose fibres had a similar development during the DSC, when there was a gradual release of heat. At temperatures around 330–440 °C, a development in heat occurred due to the pyrolysis of cellulose. In MW, there was minimal heat fluctuation; a small amount of heat was released at 400 °C due to the degradation of a resin binder contained on the surface of the fibres.

3.4. Determination of the heat of combustion

The test was performed pursuant to the EN ISO 1716 standard. The value determination was performed using a semiautomatic device, IKA C 200, under standard laboratory conditions. From the selected fibre samples, the quantity required to determine the 5 combustion heat values of the individual fibre samples was taken in the prescribed manner. The sample taken was pulverized into a fine powder using a friction bowl. To determine the individual combustion heat values, an amount of 0.4–0.8 g was taken from the powder. A precisely weighted amount of the sample was placed into a cup, which was mounted in a holder in a bomb calorimeter. The sample was connected to the combustion circuit of the holder by a cotton filament. The entire sample holder system with the sample was placed into a bomb calorimeter, which was closed and filled with pure oxygen to the inner pressure of 34 bar. The calorimeter was placed in a thermally insulated calorimetric vessel. The vessel was filled with a precisely determined amount of demineralized water. The temperature of the water in the calorimetric vessel was monitored for 3 minutes until reaching a constant value while the water was stirred vigorously. Using a combustion circuit, the specimen in the bomb calorimeter was then set on fire and the value of combustion heat was derived from the temperature change in the calorimetric vessel due to the combustion of the specimen. The result is the average of three determinations of the heat of combustion for each sample. Results are shown in Figure 6 below.

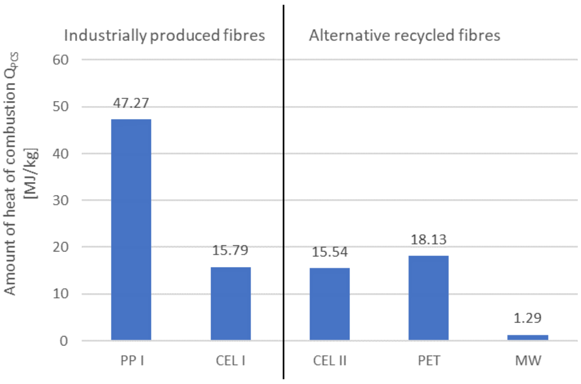

Figure 6. Graphical evaluation of the amount of combustion heat for individual fibres.

The recorded data of the combustion heat amount relatively corresponds with the results acquired by the DSC. However, in terms of generated heat, a positive result can be seen since the alternative recycled fibres show significantly lower values than traditionally used fibres. It is a comparison of PP and PET fibres. For they have a similar inner structure, cellulose fibres show almost identical values.

3.5. Scanning electron microscopy (SEM)

Three types of recycled fibres (CEL II and PET) were subjected to scanning electron microscopy to approximate the internal structure of the selected fibre types. To take pictures at extreme magnification, the Tescan MIRA 3XMU scanning electron microscope was used. The resulting pictures can be seen in the following Figures 7 to 9.

In the SEM pictures, it can be seen that the CEL II fibres (in the middle) have quite rough surface containing dust impurities and there is also a large variance in the size of individual fibres at the same time. PP and PET fibres show a smooth fibre surface with a full circular cross-section. There is a visible unevenness at the cutting point due to the mechanical separation of fibres to the required length. In both cases, these are

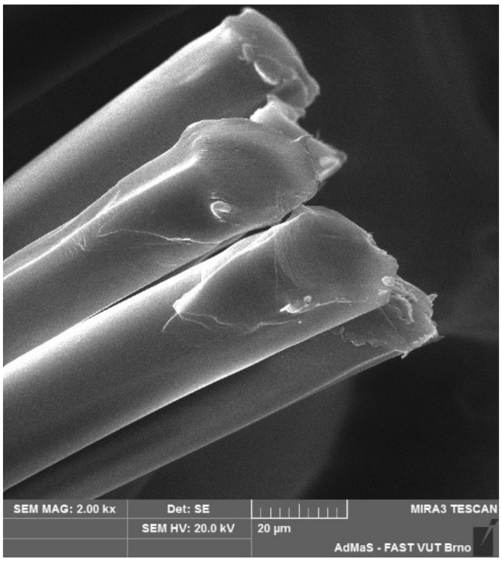

Figure 7. PP fibre structure at 2 000 magnifications.

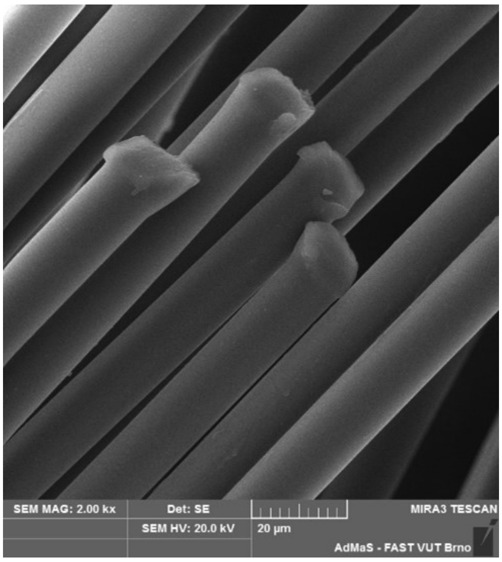

Figure 9. PET fibre structure at 2 000 magnification.

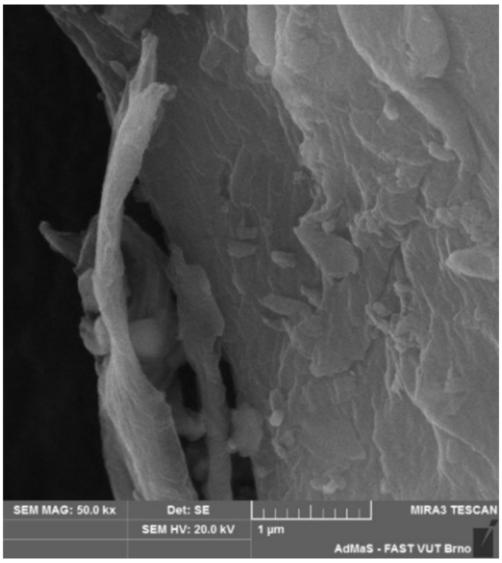

Figure 8. CEL II fibre structure at 50 000 magnification.

fibres with a fixed thickness that were produced by the extrusion of microfibre, which is then divided into the required length. PP and PET fibres can be visually compared using pictures since the magnification for both types is 2 000. The PP fibres are significantly thicker compared to the PET and at the same time significantly more damage to the cross-section of the fibre is visible when cutting to the required length.

4. Conclusion

Fibres suitable for production of cementitious composites were chosen from microfibres, which predetermine their suitability in specific applications by their geometry. The emphasis was placed on the selection of fibre from secondary or recycled raw materials since this is the direction that the construction industry will follow in order to maintain sustainability. Five types of fibres were selected to determine key properties for subsequent use as dispersed reinforcement in concrete to increase its resistance to high temperatures. The following sub-conclusions were made:

- Selected fibres are 3–36 micrometres thick, while the finest fibres are made of mineral wool, PET and cellulose CEL II (8–20 µm). The thickness of PP and CEL I is the greatest (20–36 µm).
- Fibres from recycled paper CEL II are different. It is rather a mixture of cellulose fibres of various thicknesses and lengths with a rougher surface. Due to the production technology of the recycled paper, the length variance is large.
- In terms of increasing fire resistance, the MW fibres from recycled mineral wool are not suitable since they are resistant to high temperatures, and they will not play their role in the given composite. They can be, however, used as complementary fibres that will improve the cohesion of concrete under thermal stress.
- PET fibres show significantly lower development of heat of combustion compared to the PP fibres; cellulose fibres CEL I and CEL II have approximately similar values of combustion heat.
- The SEM images show different fibre sizes and their rough surface in CEL II and smooth surface in PET and PP fibres with circular cross-section.
- From the results of thermal analysis point of view, it can be said that cellulose fibres are not suitable for increasing fire resistance because the pyrolysis of fibres occurs at relatively high temperatures when chemically bound water starts being released form the concrete.
- PET fibres seem to be an interesting substitution for PP fibres, even though the PET fibres melt at higher temperatures, above 200 °C, (as can be seen from

the TGA and DSC analysis performed) compared to the PP fibres but the heat of combustion for PET fibres is significantly lower than for PP based fibres.

From the data recorded for various types of fibres, it can be said that fibres from recycled paper CEL II can be effectively used as an alternative to industrially produced cellulose fibres CEL I, however, for concretes under stress of high temperatures, these fibres are not suitable on their own. PET fibres show comparable or even better results in all monitored properties than polypropylene fibres; therefore, there is a possibility of using these fibres as an alternative to PP fibres. First of all, PET fibres are not a primary product, but they are used as a recycled raw material. For an effective use of fibres in terms of improving mechanical and fire-resistant properties of concrete, it would be appropriate to focus on a combination of different fibre types. It can be said that it is possible to find possibilities of use between sources of recycled or secondary raw materials without consuming non-renewable resources.

For more detailed comparison of individual fibres, it is necessary to further work with fibres that have been already used in concrete or cement matrix and monitor properties of the entire system of composite material as a whole.

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