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ORIGINAL PAPER

Efficient Fabrication of Sustainable Building Products from Annually Generated Non-wood Cellulosic Fibres and Bioplastics with Improved Flammability Resistance

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Abstract

Cellulosic fibres retrieved from annual agricultural by-products offer diverse advantages when applied as a main ingredient in biocomposite building materials. Within this paper work, the application possibility of non-wood straw fibres in innovative building products is highlighted. Fabrication efficiency is reached here through reducing the number of industrial processes and additives needed to manufacture the final biocomposite products. The natural mineral contents of the straw selected (rice straw) were investigated at 20% fibre load as active flame-retardant fillers in combination with two types of bio-based synthesized thermoplastics [poly-lactic acid (PLA) and Lignin]. Flammability behavior and morphological examinations of the resulted building materials were tested. Through post-fabrication techniques including vacuum thermoforming processes, a variety of cladding panels with different architectural designs were achieved. The applied fibres were not chemically treated. Instead, the fibres were mechanically densified, maintaining the inner natural minerals contents. The results have shown promising possibility of applying straw fibres as partial replacement of classic flame-retardants especially in combination with bioplastics. The straw based green biocomposites were proved to offer high ecological, economical and aesthetic input in the building industry.

Keywords Natural fibre reinforced polymers · Bioplastic · Flammability · Fabrication · Design · Cladding

Introduction

Plastics' applications in building industry in Europe forms the second largest plastic market after the packaging industry, of which the PVC ranks the highest consumed type [1]. Globally, plastics perform around 29% from the total chemicals applied in construction [2].

There are several definitions for bioplastics. A bioplastic can be a biomass based polymer. Also fossil based but biodegradable polymers are defined as bioplastics according to the European Bioplastics e.V.

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Bioplastics emerged within the 1980s, depending on the concept of having degradable plastics that can offer another disposal option depending on biodegradability [3]. The applications were since then mostly directed towards the products' types of short life-time applications including mainly the packaging industry. This concept however have elaborated with time, even after technologically manipulating the degradability option to the fossil-based plastics. The interest to replace fossil-based plastics with bio-plastics based on renewable resources became highly increasing, especially when the bioplastics produced were meant to be more durable and not only usable in short-life time applications. This is due to their lower carbon footprint in comparison to fossil based ones.

Biocomposites is a term that describes the combination of two components: a fibre and a matrix, where at least one of them is a biomass-based component. When the two components are biomass-based, then the biocomposite is considered a green biocomposite. In both cases those biocomposites can also be referred to as natural fibre reinforced polymers (NFRP).

Applying pure bioplastics in the building sector is highly beneficial when considering the wide range of forms and colours that can be easily reached in comparison to wood and concrete. Despite of this, bioplastics are currently not widely applied in architectural applications due to their relatively high price and low flame retardancy as is the case with all plastics.

Interest in improving the flame retardance of natural fibres has been widely experimented and concerns were always raised on the possible harmful effect of the additives applied [4]. Researches have mainly concerned among other treatments the application of mineral based additives especially phosphorous based ones to improve the flame retardance of natural fibres and biocomposites [5–7].

Flammability behavior improvement of biodegradable polymers like plasticized starch thermoplastics has also been investigated and was seen to be relatively easier to improve in comparison to the classical petro-based polyolefines by adding < 10% of ammonium polyphosphate [8].

In this paper, a recycled annual natural fibre with high mineral content—namely straw fibres—has been applied as an eco-filler to improve the biocomposite's flammability retardance. This is a novel approach that was not directly addressed before as presented here and is investigated as well to reduce the final material's cost. In this case the bioplastic will be partially replaced by the cheaper straw fibres and will be more efficient for wider economic as well as ecologic applications in the building industry.

Straw, the stem of cereal plants that remain after the seed needed for nutrition is removed, composes about half the total dry weight of the crop [9]. Straw, reed and other non-wood fibres are annually renewable unlike wood. They are worldwide available, bio-degradable and have the lowest cost in comparison to all cellulosic fibres available in the industrial fibre market stock.

Since the total amount of cereal production in 2016/17 is approximately 2577 million tons [10], then the amount of available straw fibres worldwide is renewable enough to be applied in this sense as here experimented to improve the green biocomposites characteristics and decrease their final price. Rice straw has the highest silica contents within all straw types reaching until 20% after [11, 12], followed by wheat straw with high silica contents as well ranging from 4 to 10% while wood in comparison has < 1% silica contents [13]. Accordingly rice straw (RS) was selected to be applied in the experimentations. These investigations were applied to prove the possibility of applying this available renewable ligno-cellulosic resource as a main ingredient in NFRP products of high value in the building industry. This offers accordingly different solutions in managing this biomass by directing it to this specific industry instead of being burnt in open fields as still occurs in varies areas worldwide.

Since the further goal of the present paper is to investigate the possible applications of the developed straw-based green biocomposites in architecture, extrusion process took place of the pre-compounded straw-bioplastic pellets, then a series of vacuum thermoforming experimentations were applied using the extruded biocomposite panels to produce a variety of designs that can be applied as interior cladding systems in architecture. Depending on the post fabrication techniques, acoustic absorption functions as well as thermal insulation properties could be induced and reached according to the required needs.

In the following experimental section, two green biocomposites based on two bioplastics [poly-lactic acid (PLA) and Lignin] and un-treated rice straw fibres at 20% fibre load were produced (RS-PLA and RS-Lig.). A third biocomposite based on the same un-treated cellulosic fibres with the same 20% mass load and a petro-based polyolefine [polypropylene (PP)] was prepared for comparison (RS-PP). The fibre samples were analyzed before being compounded to determine the inorganic mineral contents. The chopped straw fibres were compounded with the three selected thermoplastic polymers without previous chemical modification. The natural compatibility between the fibres and the matrices were observed through SEM analysis and the effect of the natural mineral contents of the selected cellulosic fibres on the flammability behavior for the three biocomposites were studied.

Experimental Section: Materials and Methods

Straw Fibres Preparation

Rice straw applied was purchased from North Africa and was supplied in a dry de-baled raw form without any treatments. Fibre lengths upon receipt lied between 70 and 120 cm.

The straw samples were chopped using Pulverisette 25/19 fibre-chopping machine from Fritsch GmbH using the integrated chopping head sieves to prepare the fibres with lengths ranging from 0.5 to 2 mm. The fines were not sieved on purpose since it has the highest silica fraction within the whole straw [14].

Straw Inner Humidity Assessment

The inner humidity of the straw samples was measured according to the American Society of Agricultural and Biological Engineers Standards (ASAE S358.2, 2006).

The chopped straw samples were weighed before and after their dehydration for 24 h within a vacuum oven at 105 °C. The inner humidity of the samples ranged between (6–7%) and this ratio was seen as acceptable for mixing. De-hydration processes at 80 °C for 8–12 h prior to mixtures reduced the inner humidity temporarily, but the same inner relative humidity returned rapidly to the original value. The samples were kept in a desiccator with a drying agent to prevent further humidity increase.

Straw Fibres Analysis

The RS fibres were chopped and burnt at 550 °C to prepare the rice straw ash samples and the inner chemical inorganic components of the rice straw ash samples were analyzed to measure the silicon component load. The rice straw ash weighed 14.8% of the original straw weight. In the selected rice straw sample silicon contents were around 73.3% in the rice straw ash, meaning around 11.1% of the whole rice straw weight. In Table 1 the inner non-organic components of the analyzed ash are indicated.

This high silicon content's reflection on the flammability attitude of the developed rice straw biocomposites is thoroughly analyzed in the characterization section.

Binders Applied

Two types of bioplastics were applied (PLA and Lig.) and a petro-based thermoplastic was applied (PP) for comparison. Poly-lactic acid (PLA) 80 150 D was supplied from Nature Works LLC, USA and Lignin bioplastic Arboform® was supplied from Tecnaro GmbH, Germany. Conventional polypropylene (PP) granulates were purchased from a plastics supplier in Germany.

Lignin is here applied as a thermoplastic, against its known nature as a thermoset polymer, after being synthesized and modified by plasticizers to act as a thermoplastic by Tecnaro Company—the supplier.

Biocomposites Processing

The fibers (RS) and the three chosen polymers (PLA, Lignin, PP) were separately compounded with 20% of RS fibre load of the whole mixture in a twin screw extruder PTW16 from Thermo Electron to produce compounded pellets then injected molded using an injection molding machine Thermo Scientific HAAKE MiniJet II then cut to smaller sample sizes of $70 \times 10 \times 2$ mm of volume 1400 mm^3. The three biocomposites are two green biocomposites referred to as RS20%-PLA and RS20%-Lig. and a biocomposite with a fossil-based organic thermoplastic polymer PP that was taken as a reference, referred to as RS20%-PP. These samples were the ones used for characterization, flammability and morphological examinations.

Biocomposites Extrusion and Vacuum Thermoforming

To evaluate the applicability of the developed materials in the building industry, one of the compounded pellets' types—namely RS-PLA—was extruded using a single screw extruder Alpha 45-25 B from Cincinnati Extrusion GmbH between 160°–180° with 20 rpm. Rectangular planar profiles were applied to extrude plates of 2 and 4 mm thicknesses, supported by the company think-blue in Germany.

Different molds were manufactured according to a number of designed geometries. Molds out of gips, clay, PU foam, wood and rubber have been trialed and applied. The molds were manufactured using different fabrication processes including casting, CNC drilling and laser-cutting based on CAD—models. The extruded biocomposite panels were preheated up to 500 °C, then placed on the pre-fabricated molds in a vacuum press machine from Barth. Variations in the preheating temperatures, heating time intervals as well as different vacuum pressing time intervals were investigated [15, 16].

The developed materials offer a wide range of possible applications as building materials and as possible replacement to the known wood polymer composites (WPCs) especially in interior applications. The disadvantages of WPCs are mainly due to the main ingredient wood, which is a slow renewable resource that is not available in many plots worldwide which is opposite to the case of agro-fibers' renewability and availability. Furthermore, within WPCs other than the UV additives, extra flame retardant additives are needed to be added. This can be completely or partially overcome in the case of cereal straw's appliance due to its natural high silica contents as here investigated. A wide range of geometrical variations can be reached through the application of plastics in architecture. Accordingly the architectural possibilities of the developed materials using thermoforming processing techniques are diverse. In Fig. 1 a number of thermoformed straw-based green biocomposite materials with different designs are illustrated. The thermoformed panels can be applied as interior cladding systems or as covering surfaces for sandwich panels, whether for acoustic absorption or thermal insulation performances.

Table 1 The average inner chemical composition of the inorganic ash of the rice straw samples

Mineral	Al	Ca	Fe	K	Mg	Mn	Na	P	S	Si	Zn
mg/kg	2.1	18.8	1.6	80.2	6.9	2.9	1.9	6.5	4.6	343.8	70.1

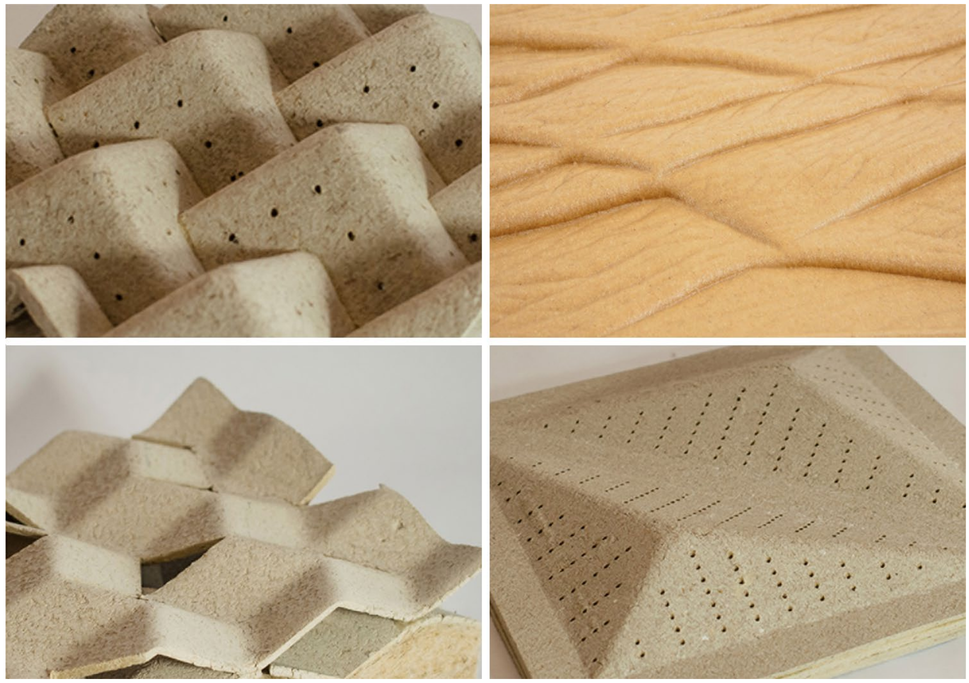

Fig. 1 Geometrical variations and designs of thermoformed straw-based biocomposites (Photo: Dahy)

Characterization Methods

Density

The densities were measured according to DIN 53 479 or DIN EN ISO 1183-1 at 23 ± 1 °C.

Morphology Studies

RS green biocomposites: RS 20%-PLA and RS20%-Lignin were examined at their fracture and top surfaces using a scanning electron microscope (SEM).

Flammability Behavior

The test results were both quantitatively and qualitatively analyzed. Tests took place on the 70 × 10 × 2 mm samples. Results were measured in seconds, which were evaluated according to the Standard UL 1694 test conditions for flammability of small polymeric component materials. Applicable sample volume to be tested through this test was limited to 2500 mm^3 to describe the material class to which the manufactured materials belong to. Observations of the fire behavior were also recorded. The test results cannot be taken as an exact evaluation of the material fire class that can be directly applied in building construction or as a finishing or cladding material, but rather considered as a general evaluation of the flammability behavior of the examined materials. To determine the exact flammability behavior of the materials to be applied in the construction field, DIN 4102 or SBI (single burning item test) after DIN EN 13501 should be applied instead.

Fire Test Procedures, Conditions of Testing and Evaluation Method

Apparatuses Used

A laboratory fume hood with volume of ca. 1.125 m^3 was applied to provide enough oxygen for normal combustion. A laboratory small burner of approx. 60 mm length, 0.5 ± 0.1 mm inner diameter and approx. 0.8 mm outside diameter was applied. The burner was connected with a gas supply and had a flame height of max. 12 ± 1 mm. A laboratory ring to enable positioning the burner in the required angle and height was used and a digital time device with 0.5 s accuracy as well as a cotton pieces of 50 × 50 mm was placed under the hanged specimen as an indicator.

Test Description

The testing was applied in normal room temperature (approx.23 °C) and without further material drying, hence normal inner humidity was present. Each sample was fixed with a clamp in a vertical position above the indicator by 200 ± 5 mm. The lab-burner was tilted at the edge of the sample at 45 °C with an average vertical distance of the flame of approx. 8 ± 1 mm from the specimen's end. According to the specimen's volume, the time of flame application (t_a) was defined according to the standard. Since the sample's volume was 1400 mm^3, the (t_a) was set to be 20 s. After the flame application for the specified time interval, the burner was withdrawn for approx. 150 mm away and the timing after flame application till the flame ceased was recorded (t_{b1}). In some cases the flame ceased shortly after flame application, so the flame was directly re-applied for the same time interval that was applied at the beginning. After the second flame application, the time was again recorded from the second flame application till the second flame ceased as well and stopped flaming or glowing, which is the after flame time that was recorded as well in seconds (t_{b2}). The same previous step was repeated when the sample was not yet consumed and the after flame time—after the third ignition—was recorded in seconds (t_{b3}).

Evaluation Method

The test was recorded both qualitatively and quantitatively according to the following criteria:

Qualitatively

- t_a = flame application time according to the sample's volume (20 s)
- t_{b1} = after flame time, after first flame application
- t_{b2} = after flame time, after second flame application
- t_{b3} = after flame time and glowing time, after third flame application.

Quantitatively

- Whether or not specimen pieces melt and dropped on the cotton indicator and caused its ignition.
- Whether the sample has totally been consumed or not.
- Notes of visual observations concerning unusual fire behavior or flame traces on the samples.

Results and Discussion

Density

Applied chopped straw in its raw form is of average 40–100 kg/m^3 [17]. RS 20%-PP had the lowest density within the developed biocomposites, while RS20%-Lig. and RS 20%-PLA were of much higher densities as displayed in Table 2.

Table 2 Densities of the developed biocomposites

Biocomposites' groups	RS 20%-PLA	RS 20%-Lig	RS 20%-PP
Density (g/cm^3)	1.2	1.3	0.9

Morphological Examination

SEM scanning electron microscopic examinations at the surfaces and the fractures of the torn-out samples resulted from the tensile test helped in better understanding the relationship between the raw milled straw and the bioplastics in the case of RS20%-PLA and RS20%-Lig. This examination helped to distinguish how homogenous and compatible those biocomposites are and how this might link with the differences in their flammability behaviors.

The samples' examination photos of the top surfaces and the fracture surfaces are illustrated in Figs. 2 and 3 respectively.

Through the SEM photos, it is clear how the RS20%-Lig. has un-sealed surface as seen in (Fig. 2) in comparison to the RS20%-PLA, while compatibility of RS20%-Lig. is recognized to be much higher than the RS20%-PLA in the fracture surface morphologies' comparisons (Fig. 3). The strong attachment between the RS and the lignin matrix is highly recognized through the illustrated microscopic examination. The effect of this compatibility was examined as well according to the differences of the flammability behaviors of the developed biocomposites.

Flammability Behavior: Results and Evaluation

The effects of the high silica contents present in the applied raw rice straw on the flammability attitudes of the developed biocomposites were evaluated as presented in Table 3. The non-presence of additives enabled the objective comparison to take place, eliminating the presence of extra factors that could have caused evaluation mistakes.

RS20%-PP was completely melt down and kept burning without self-extinguishment till the clamp. The flammability behavior of RS20%-PLA was much better than that of RS20%-PP. RS20%-Lig. flammability resistance behavior was the most successful in the three developed biocomposites, probably due to the special relationship between the silica and straw lignin present together in a special combination in the RS structure [19]. This increased the compatibility and gave the silica the chance to precipitate properly and homogenously through the composite's structure, having enough chance to optimize the fire-resistance effect as recorded. This aspect is important in the building industry and architectural applications.

Conclusions

Rice straw was bonded in its raw form with a mass load of 20% with PLA and lignin bioplastics. The effect of the natural existing silica in the rice straw on the final developed green RS—biocomposites were evaluated in respect to the

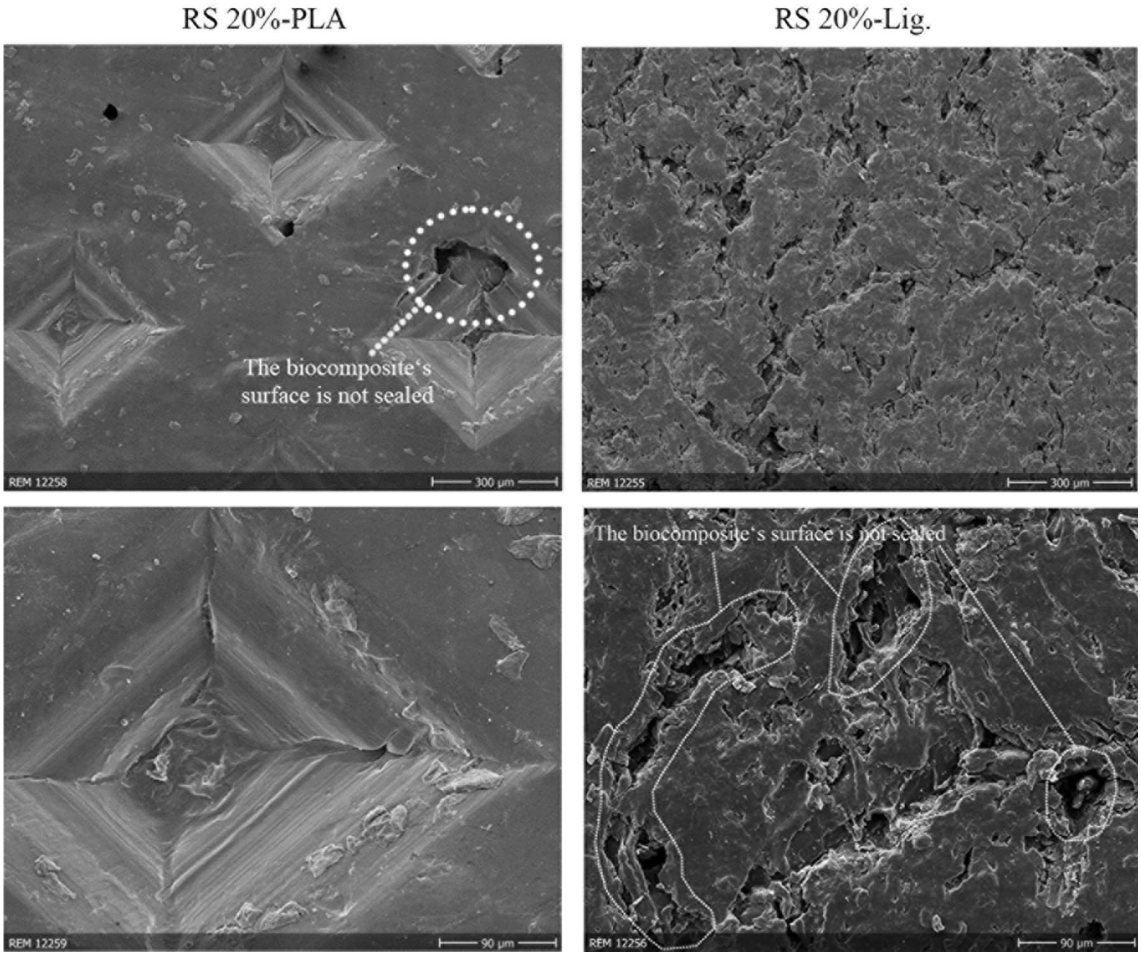

Fig. 2 Comparison between RS 20%-PLA and RS 20%-Lig. surface morphology using SEM-microscopic analysis

density, morphology and flammability behavior. In addition, the possible architectural applications of these materials were accordingly illustrated after being vacuum thermoformed according to different custom designs.

Concerning homogeneity and compatibility, RS 20%-Lig. biocomposite has shown higher compatibility than RS20%-PLA. This was clear through both the morphological SEM examination and the fire-test results. RS 20%-PLA has shown better fire retardance than RS 20%-PP biocomposite. This indicates the possible application of certain types of highly filled silicate agro-fibres as a direct replacement either fully or partially of mineral-based flame retardants in plastics, especially bioplastics. Since flammability behavior in building materials plays a crucial role in applying the materials in certain applications or building categories, the importance of this novel approach arises strongly. The problems linked with halogen-based flame retardants force researchers to search for more environmentally-friendly alternatives. That was a good reason that mineral based flame retardant additives were the possible reasonable alternative. In this paper, applying a natural fibre of high mineral contents as a natural available flame retardant dependent on the natural compatibility between the cellulosic fibres and the bio-based plastics applied was examined.

Generally it can be concluded that RS20%-PLA and RS20%-Lig. can partially replace available WPC materials that are mostly based on fossil-based plastic materials and are widely spread in architectural applications. The ecologic, economic and design advantages of these developed biocomposites are multiple, which promote them to be widely applied in the building industry.

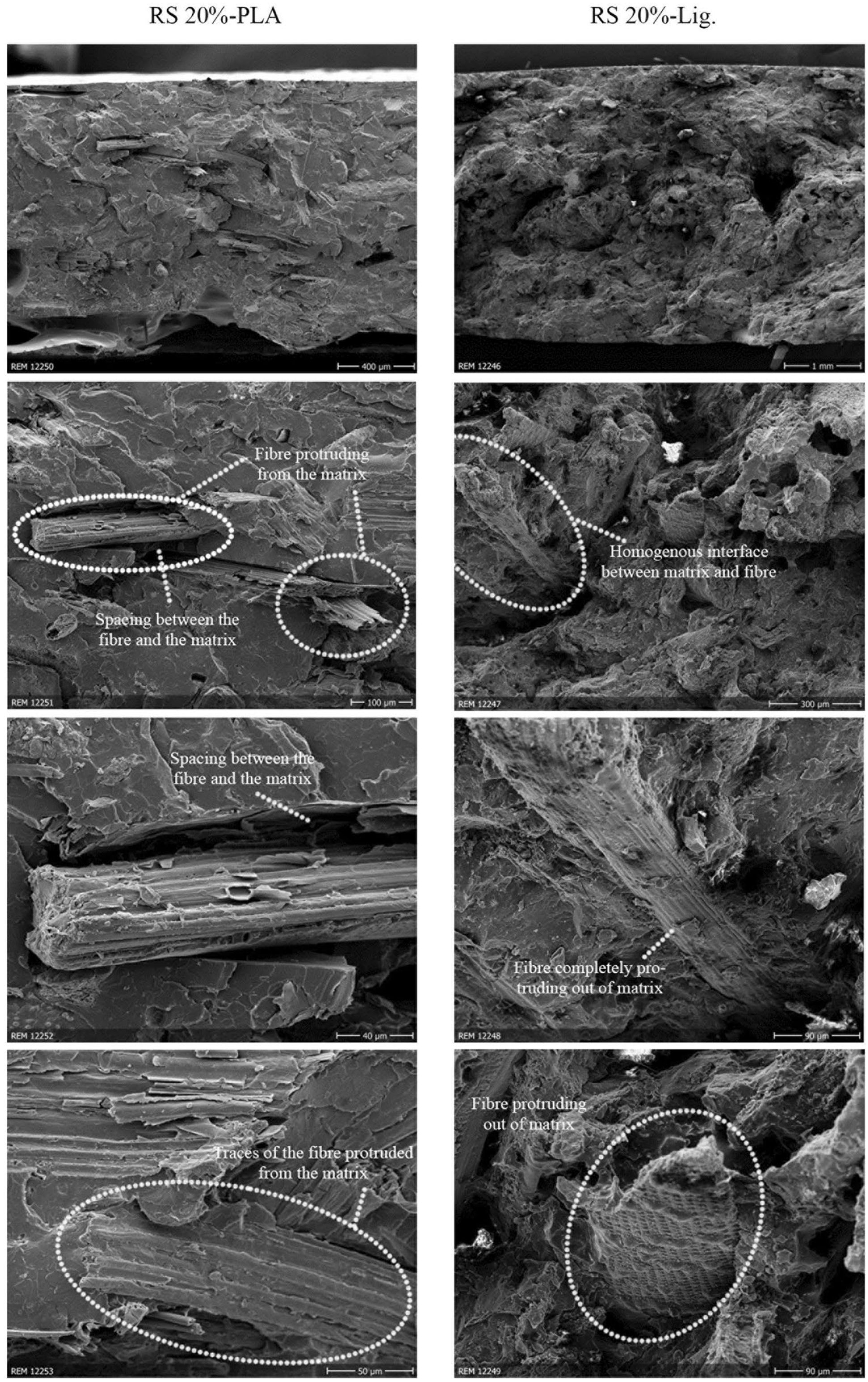

Fig. 3 Comparison between RS 20%-PLA and RS 20%-Lig. fracture surface morphology using SEM-microscopic analysis

Table 3 Illustration of the flammability attitude of the RS 20%-PLA and RS 20%-Lig. in comparison to RS 20%-PP as a reference biocomposite after UL1694 [18]

Biocomposites groups	RS 20%-PLA			RS 20%-Lig			RS 20%-PP (Reference)		
[sec.]	t_{b1}	t_{b2}	t_{b3}	t_{b1}	t_{b2}	t_{b3}	t_{b1}	t_{b2}	t_{b3}
Spec.1	3	15	–	7	30	7	105	–	–
Spec.2	2	58	–	5	30	10	112	–	–
Spec.3	2	35	85	6	28	8	148	–	–
Spec.4	3	41	50	8	32	12	110	–	–
Spec.5	4	50	–	5	27	5	115	–	–
Cotton indicator ignition (yes/no)	Yes			No			Yes		
Complete consumption of specimen (yes/no)	2 samples: yes, 3 samples : no			No			Yes		
Notes	A part of the sample did not burn in 3 tested samples. The sample's ignition stopped after < 5 s for all samples and the second ignition stopped after < 60 s for all samples. However the developed material did not have a specific material class, but was closely equivalent to UL1694-TC-2			The first after flame time was < 10 s, while tb 1 + tb2 were together < 50 s tb 2 + tb3 were together < 60 s. In addition, the specimens were not consumed and there were no droplets on the indicator. Accordingly, the detected fire class was UL1694 SC-1			The samples were completely consumed and took > 100 s till the flame reached the clamp and the burning stopped. The samples' test results described a material, which is below standards and cannot be specified accordingly to a certain material class		

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