Research of new flame retardancy methods and mechanisms for the development of lightweight polymer composites

Új égésgátló módszerek és mechanizmusok kutatása súlycsökkentett polimer kompozitok fejlesztéséhez

OTKA FK 128352

Final research report

2018. 10. 01. – 2023. 09. 30.

Summary of results

Our research has been focused on the development of innovative flame retardant methods for lightweight yet highly flammable polymer composites, such as natural fibre-reinforced, self-reinforced and foamed materials. Special emphasis has been placed on the aspect that the newly produced polymer composites, the new flame retardant solutions and the applied production technologies follow the guidelines of green chemistry and the circular economy as much as possible. For this purpose, composite components of renewable (such as polylactic acid, natural fibers, cellulose, sorbitol, glucose, cyclodextrin) or recycled (recycled PET) origin were used, from which recyclable (self-reinforced) and / or biodegradable (natural fiber-reinforced, biopolymer-based) composites were manufactured. In the development of new flame retardant additive systems, also biobased starting materials (such as sorbitol, glucose, cyclodextrin, etc.) were processed, by-product-free addition chemical reactions (e.g. phosphorus-silane production) were preferred, and instead of organic solvents, where possible, water was used (electrospinning of cyclodextrin, flame retardant treatment of natural fibres). Novel flame retardancy mechanisms have been explored through physical and chemical modifications - such as fibre formation, surface treatment or encapsulation - of various polysaccharides and by utilising synergistic interactions between the active ingredients of biocomposites, respectively. The discovered new manufacturing methods and mechanisms can be exploited to achieve the desired property (eg. flame retardancy level) with significantly less additives (and cost) (eg. combined intumescent flame retardant additive systems, toughening of recycled PET, particle size effects). Monitoring of the applied continuous manufacturing technologies (eg extrusion foaming) using in-line applicable spectroscopic methods (NIR, Raman) was demonstrated to be useful in reducing by-product/waste generation, reducing off-line testing costs and saving human resources, thus opening up the possibility of more economical production of environmentally friendly polymer composites.

The main results of the four-year research work are classified by topic below:

Exploring structure – property relationships in self-reinforced polymer composites

Self-reinforced polylactic acid (PLA) composite sheets with special nanofibrous structure were manufactured (K. Bocz et al. Polymers 2018;10(7): 1-12.). The production of PLA microfibers, serving as a reinforcing phase, was achieved with the uniquely high yield of 40 g / h using the high-speed electrospinning process (HSES) developed by our research group. Then, our research concentrated on the marked discrepancies in the crystalline structure of PLA nano- and microfibres, achieved by different annealing strategies, i.e. heat treatment and solvent treatment. It was found that only disordered α′ crystals are formed during the conventional thermal annealing; however, the ethanol-induced crystallization favours the formation of the ordered α polymorph. We have pointed out that these differences in the crystalline structure cause significant changes in the macroscopic characteristics, e.g. heat resistance and mechanical properties, of the microfibres and their self-reinforced composites as well [3].

Development and flame retardancy of environmentally friendly polymer foams

The development and characterisation of flame retarded low density (ρ <50 kg/m^3), microcellular PLA foams prepared by supercritical CO_2 assisted extrusion have been published before the actual starting date of the project (K. Bocz et al. *Polym Degrad Stab* 2018; 153: 100-108.). In this work, the foamability of PLA was improved by the appropriate addition of reactive chain-extender and nano-sized clay mineral particles, while a new intumescent flame retardant additive system, containing cellulose treated with phosphorus and boron-containing compounds, was developed to reduce flammability of the biofoams.

In the frame of the OTKA FK research project, with the further development of the extrusion foaming technology, we have been able to produce PLA foam products with uniquely structured, lens-shaped cells (Fig. 1), which have several advantageous properties: they are elastically deformable, and have adjustable modulus of elasticity (0.1-5.0 MPa) and piezoelectric charge [22]. We have filed a patent application for the invention which, due to its biocompatibility, could be used in, inter alia, medical devices (drug pumps, artificial tissues) or disposable energy harvesting products [2].

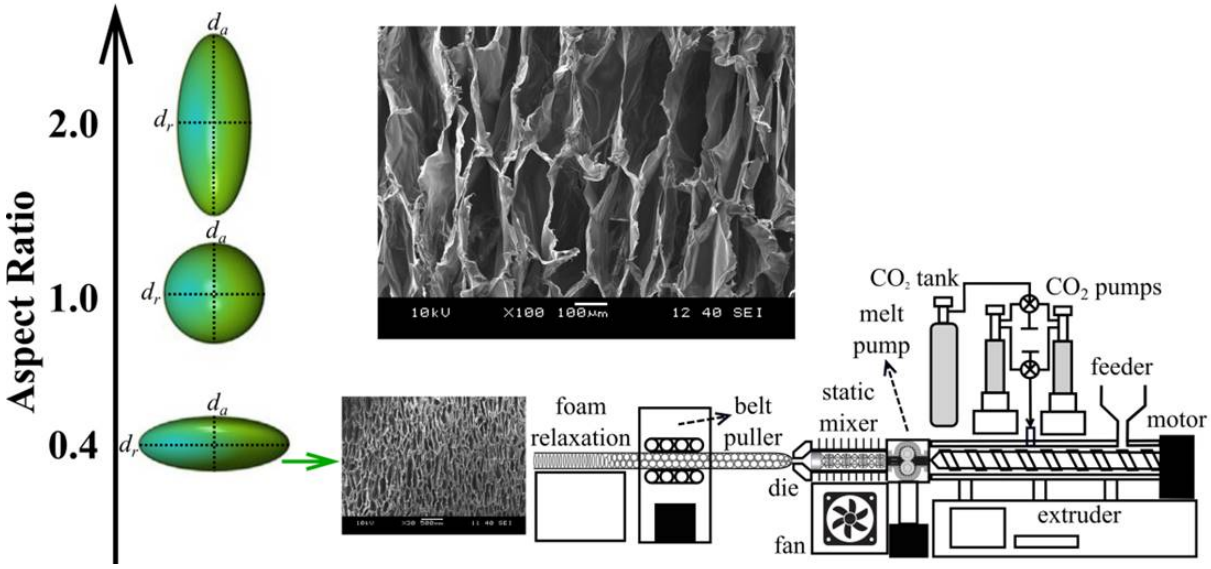

Figure 1 Manufacturing of highly flexible poly(lactic acid) foams and ferroelectrets

Value-adding recycling of secondary raw materials

The recycling of significant amounts and varying qualities of poly(ethylene-terephthalate) (PET) waste, whether significantly degraded (selective municipal waste, industrial waste, or even marine waste) is a major challenge today. Recycling usually results in significant embrittlement of the material, which limits its potential uses. The production of high-performance plastics from recycled PET (RPET) is therefore a particularly important and topical goal, which is further encouraged not only by increasingly stringent legal requirements but also by the ever-increasing price of the original polymeric raw materials.

Improving toughness of RPET

As a first step, the change in the crystalline structure of PET, being particularly prone to hydrolytic degradation, as a function of its molecular weight has been comprehensively investigated [10].

Subsequently, a new process has been developed, in which the waste PET fraction contributes to the outstanding increase of the impact resistance of the plastic product - exceeding the performance of ABS, HDPE and PA6 (30–50 kJ / m^2). At the same time, as lower amount of reactive additive is necessitated, a cost reduction of about 20% can be realised. The key to this solution is that the efficiency of the reactive impact-modifier additive can be increased through the increased mobility and reactivity of the reduced-molecular-weight polymer chains characteristic for degraded PET waste [13,26]. In this way, the technology provides a solution for the economical recycling of even highly degraded, non-marketable PET waste in a productive, continuous way (eg. injection molding or 3D printing) that was not previously typical for this material [19,24,36]. We filed a patent application for this technological process [1].

It is also claimed that the efficiency of reactive modifiers in various polymeric systems can be effectively increased by the targeted utilisation of the increased reactivity of low-molecular-weight polymer fractions, whether origination from highly degraded, unmarketable waste fractions or formed in situ during melt processing.

The significance of our subsequently published paper [17] for the research community relies mainly on the proposed significant paradigm shift from the former view that moisture clearly reduces the impact strength of PET-based blends during processing or reprocessing. As most important results, it is shown that a more than 6-fold increase of notched impact strength can be achieved only by "adding some water" to the PET/EBA-GMA system or by simply omitting the conventionally performed drying step. The evinced outstanding increase of the notched impact resistance is found to be related to the intensified PET/EBA-GMA compatibilisation reactions and suppressed crosslinking of the rubber phase.

As a continuation, this phenomenon has also been successfully exploited in increased processing temperature assisted reactive toughening of PLA [18,25].

Foaming of RPET

Our experience in the production and flame retardancy of microcellular PLA foams [28] has also been transferred to secondary polyethylene-terephthalate (RPET); low-density (ρ <350 kg / m^3) microcellular foams were successfully produced from the recycled raw material by continuous extrusion technology. The molecular weight of the degraded recyclate was effectively increased by two methods, solid phase polymerisation and the use of a reactive chain extender, to obtain a raw material suitable for foaming [5].

In our sc-CO_2 dioxide assisted foaming experiments, if the intrinsic viscosity (IV) of the recycled material was increased from 0.62 dl / g to 0.87 dl / g using an epoxy-functional chain extender, and the addition of talc effectively aided nucleation and stabilisation of the cells, RPET foam products with a density of even less than 150 kg / m^3 were obtained. We found a strong correlation between the apparent density of the foams and the near-infrared (NIR) spectrum of the foamed RPET samples, which allowed the in-line, rapid, and non-destructive characterisation of product quality. Accordingly, NIR spectroscopy has been found to be a suitable method for in-line monitoring of product quality during extrusion foaming of recycled PET, which is particularly prone to quality fluctuations [12].

Flame retardancy of RPET

The flame retardancy possibilities of RPET were also investigated. By combining aluminium-alkyl-phosphinate and montmorillonite clay mineral, nanocomposites with reduced flammability and excellent mechanical properties were produced. The applicability of the value-added recycled material in the electric or electronic fields was demonstrated by injection moulding of television housings [6].

Using the optimised formulation, flame retarded microcellular foam products (ρ = 200–350 kg/m3) from bottle-grade RPET have also been successfully produced by sc-CO_2 assisted extrusion [16] [29] and by batch foaming technology as well [23, 35]. The fine microcellular

structure and high cell density, obtainable by the batch foaming procedure, allow homogeneous distribution of FR additives, and thus only moderate increase in flammability was observed for the high-porosity (>75%) foams compared to the corresponding bulk materials, as characterized by similar limited oxygen index (LOI) values. The mechanical performance of the flame retarded RPET foams was found to be primarily determined by the apparent density and less affected by the presence of FR additives. Due to strain-induced crystallization occurring during cell growth, the RPET foams are highly crystalline ($\chi > 25\%$) which leads to increased thermomechanical resistance compared to unfoamed references. The flame retarded RPET foams are therefore promising to be used primarily in transportation or construction as lightweight heat insulator or sandwich core materials.

Flame retardancy of biocomposites – development of new additives and exploring new mechanisms

New FR additives

Multifunctional additives have been developed which – in addition to their flame retarding effect - have reinforcing, heat stabilising and / or compatibilising effects as well. These additives can be used to reduce the amount (and cost) of additives required for flame retardant biocomposites.

New phosphorus-containing silane (PSil) adducts were prepared by addition reactions of phosphorus-containing polyols (such as ethylene-glycol-phosphate, glycerol-phosphate, Exolit OP560) and 3- (triethoxysilyl)-propyl-isocyanate. The products were used as reactive surface treating agents for the flame retardancy of cut flax fibres as well as cyclodextrin microfibres. Thermogravimetric studies have shown that treatment with new PSil greatly promotes carbonisation of biofibres without reducing their thermal stability [30].

During the research period, the use of various carbonising agents of renewable origin were tested with the aim to provide environmentally benign flame retardant formulations, among others for poly(lactic acid) (PLA) [15,20,38,39,40], polybutylene-succinate [34], epoxy resin (EP) [37,38,40] and polypropylene (PP) [14], respectively.

Sorbitol and glucose-based bioepoxy resins were synthesized and then tested for their use as flame retardant components (such as efficiency, compatibility with biopolymer matrices, water sensitivity, stability, price). Processes for microencapsulation of ammonium-polyphosphate (APP) and melamine-polyphosphate (MPP) with bioepoxy resins have been elaborated. The microencapsulated additives with the optimized composition have proven to be easy to handle (combined), waterproof, and effective flame retardants in PLA matrix [7,27] and in PP matrix as well [14]. The improved flame retardant performance is attributed to the effective interaction between the APP core and the readily available carbonising bioepoxy shell, which promotes the formation of increased amount of char accompanied with improved heat protecting and barrier efficiency. In addition to significantly improving flame retardant

properties, they provided better filler-matrix interaction, increased modulus, and better water resistance compared to the neat additives.

In accordance with the work plan, complexation of Mn, Zn, Fe, Cu, and Ni metal ions with diaza crown ethers were successfully accomplished. The complexes were then embedded in PLA matrix and the thermal and flammability properties of the thus obtained PLA composites were analysed. According to thermogravimetric analyses and pyrolysis combustion flow calorimetry results, among the investigated metal ions, Fe has the greatest char promoting effect in PLA. However, besides catalysing the charring of PLA, the Fe-crown ether complex was found to noticeably accelerate the molecular degradation of PLA during processing, causing applicability issues.

Therefore, as a new research direction, alginate gels, derived from natural source, with diverse crosslinking metal ions were prepared and investigated as potentially synergistic co-additives hereinafter. The Na-alginate was also reactively modified with a phosphorous silane (PSil) compound, and then coagulated by calcium ions (Fig 2.). The obtained Ca-PSil-Alg additive was characterized by spectroscopic and thermoanalytical methods, while its flame retardant effect was evaluated after embedded in PLA matrix besides 15% APP. It was found that using only 5% of the newly produced alginate-based additive the LOI of the PLA composite increases from 26% to 34%, while the THR reduces by nearly 50%, in addition to the formation of a significant (~40%) carbonaceous residue. The outstanding flame retardant effect is explained by the effective self-charring of the modified alginate catalysed by P, as well as by the ceramization effect caused by Si, which together contribute to the formation of a compact, thermally and mechanically resistant fire protecting layer. Main results have been presented in international conferences [32,33], the corresponding research paper is in preparation.

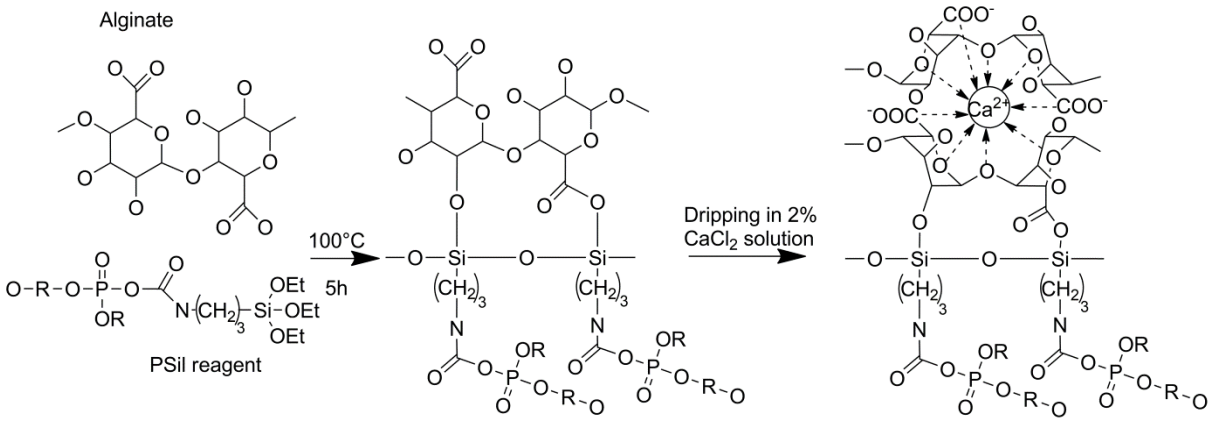

Figure 2 Preparation of Ca-PSil-Alg additive

New FR mechanisms

The role of the physical form of a polysaccharide-type carbonising component on its effectiveness in an intumescent flame retardant system has been firstly investigated. Microfibrous structures were prepared from the aqueous solution of (2-hydroxypropyl)-β-

cyclodextrin (CD) by electrospinning process. It was shown that the polymer-like supramolecular structure can greatly increase the flame retardant efficiency of cyclodextrin in intumescent flame retardant systems. It was found that the advantage of the special microfibrous structure of the oligosaccharide type carbonising agent lies in the effective interaction with APP and the formation of a carbonaceous protective layer with increased thermal and mechanical resistance. The fact that only by changing the aspect ratio of CD significant improvement can be achieved in both the flame retardant effect and the mechanical properties is a new finding [11].

As a continuation of this research, the flame retardant performance of the electrospun CD was further enhanced by reactive surface modification with the phosphorous silane (PSil) compound. The beneficial effect of the PSil-treated CD microfibre relies mainly on its enhanced physical and chemical interaction with APP and its increased charring ability and thermal stability, which contribute to the formation of a fire protecting char of increased structural integrity [15,31,38,40].

In the frame of an international collaboration with the Bundesanstalt für Materialforschung und -prüfung (BAM) organisation in Berlin (Germany), the high-surface-area electrospun cyclodextrin short fibers and nonwovens were reported as effective fillers in flame retarded epoxy composites as well [37].

MPP was combined with various types of cellulosic fibres, differing in fibre length, to obtain intumescent flame retarded PLA composites. It was found that the formation, swelling ability, structure and mechanical resistance of the fire protecting char is noticeably affected by the length of the used cellulose fibres and thus it basically determines the efficiency of the flame retardant system. Structure-property relationships were evaluated using morphological, thermal, flammability and mechanical analyses. Cellulose fibres with an average length of 30–60 μm were found to contribute the best to the formation of an integrated fibrous-intumescent char structure with enhanced barrier characteristics [21,39].

The structure-property relationships of flame retardant multilayer biocomposites with phosphorus- and nitrogen-containing compounds were analysed by experimental design and multivariate evaluation methods. Positive interactions have been shown between the flame-retardant-treated composite phases in terms of several flammability (eg oxygen index, ignition time, maximum of heat release rate) and even mechanical characteristics (tensile strength). Our results demonstrate that the combined flame retardancy approach of multilayer biocomposites, i.e. the balanced distribution of phosphorus-containing additives between the matrix and reinforcement phases, results in increased mechanical performance and economic advantage as well [4].

Our new insights into the manufacturing technology of biocomposites (compression versus injection moulding) and our results in the field of Raman signal-based monitoring and control of composition and structure have been reported in further impact factor journals [8,9].

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PCT patent application filed at: Hungarian Intellectual Property Office,
application number: WO/2022/112807

[2] Vadas D, Igricz T, **Bocz K**, Marosi G
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[13] **Bocz K.**; Ronkay F. ; Decsov K. E.; Molnár B.; Marosi G.:
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Main scientific indicators:

patent applications: 2

international journal articles: 22

PI highlighted (first or last) author**: 17**

Q1: **15**

summa impact factor: **90**

national journal article: 1

international conference presentations: 13

PhD supervision: 3

PhD degree: **1** (Dániel Vadas, 2021)

PhD thesis submitted **1** (Decsov Kata Enikő, successful first defense: 08. 09. 2023.)