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Processing of Latvian peat and waste coffee as a biocomposite material for the oil spill collection

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Abstract. There is a growing interest in adsorbents of natural origin that are renewable, effective, and able to treat water contaminated by oil products. The current paper investigates a novel bio-based 'peat - spent coffee grounds' SCG-HP bio-based composite pellets as a perspective adsorbent for spilt oil products. The preparation and characterisation of SCG-HP bio-based composite material in pellet form is described. This research used homogenised peat (HP) as an efficient, natural binder. The SCG in different proportions (from 12 wt% to 50 wt%) with HP were used for the different types of SCG-HP granulated sorbents. The granule size obtained ranged from 2 to 6 mm with a total porosity of 56–61%. The sorption of the test oil (fresh engine oil Pilot 10W-40 SJ/CF) was investigated. Sorption studies showed maximum adsorption (capacity) from 90 to 125 wt% for SCG-HP granules.

Key words: spent coffee grounds, adsorption, peat bio-based composite, oil spill, sustainable production, waste recycling.

INTRODUCTION

There is a significant focus within the scientific community on using by-products to develop novel materials. One of specific but prospective organic waste is coffee waste from coffee making. Recent data show increased consumption of used coffee grounds (SCG), which are the waste from the coffee brewing process. Around 6 million tonnes of SCG were generated in 2020–2021 (Hu et al., 2022). Coffee residues are the largest waste product because they are a highly sought-after commodity on the international

market, resulting in large quantities of coffee waste in the form of SCG (Kim & Kim 2020).

Despite the specific properties of SCGs, there are many potential applications for SCGs and SCG-based products. Table 1 summarises some of the promising application fields of SCGs in environmental areas and for developing new materials.

Table 1. Concise overview of recent research on applications of Spent Coffee Grounds

	SCG application area	References
1.	SCG as an additive for food (bakery) products	(Benincá et al., 2023; Cavanagh et al., 2023)
2.	SCG for obtaining electrospun composite nanofiber	(Rubio-Valle et al., 2023)
3.	SCG for graphene oxide production	(Challa et al., 2023)
4.	SCG application in fluorescent chemosensors	(Jeong et al., 2023)
5.	Development of nanostructured and KOH-activated carbon for energy storage application.	(Padilla-Martínez et al., 2023; Sangprasert et al., 2022)
6.	Microporous carbons from SCG for selective extraction (explosives removal) from water samples	(Charmas et al., 2022)
7.	Biochar obtained by carbonisation of SCG for an energy storage device	(Andrade et al., 2020)
8.	Ceramic materials with clay and SCG	(Kłosek-Wawrzyn et al., 2023)
9.	Films from lignocellulosic fibers of SCG	(Bhattarai & Janaswamy, 2023)
10.	SCG for bio-based phase change materials for thermal energy storage	(Jin Ong et al., 2023)
11.	Enhancement of building materials (concrete)	(Roychand et al., 2023; Saeli et al., 2023)
12.	SCG valorisation in biorefinery applications	(Lauberts et al., 2023; Sharma et al., 2021)
13.	Hydrogen production from SCG	(Bekirogullari, 2020; Rodrigues et al., 2022)
14.	SCG as a fuel for small boilers	(Kang et al., 2017)
15.	Microalgae growth on SCG substrate for biodiesel production	(Rosmahadi et al., 2022)

Using SCG as an adsorbent for petroleum products is considered an innovative application, as evidenced by existing research in this field (Lee et al., 2022; Shi et al., 2023).

Peat as a binder is also novel in the context of use with SCG. Peat is a complex system with a wide variety of properties. Intense mechanical action can be used to alter the physicochemical properties of peat and the properties of the macromolecular compounds it contains (Mazlan et al., 2023). Mechanical activation of peat by dispersion increases specific surface area and the opening of closed pores. The deformation of the peat components causes changes in the interatomic and intermolecular bonds, leading to their weakening and, in some cases, their destruction, resulting in the formation of free radicals.

The mechanical activation of the peat is carried out in a disperser. The treatment can be carried out without reagents or with their addition for different purposes. For example, adding solid NaOH/KOH promotes the release of humic substances. It increases the release of soluble peat fractions such as polyphenols and polysaccharides and its binding capacity (Klavins & Purmalis 2013).

MATERIALS AND METHODS

The SCGs used in this study were obtained from local coffee shops in Riga, Latvia. The collected SCGs were dried at 105 °C until constant weight; otherwise, the samples tended to mould and then sieved through a 0.250 mm sieve. The sieved SCGs were stored in polyethene bags until use and did not undergo any physical or chemical pretreatment before use. SCG was characterised as having a broad particle size range from 20 μm to 30 mm, being composed of fibre (> 50%) and lignin with high molecular composition and having a high surface area (7.5 m^2g^{-1}).

The binder used in this study is ZTK low-type peat (Lielvārde, Latvia). The material was subjected to metal analysis to rule out the presence of heavy metals. ZTK was mixed with distilled water in proportions 1:2, taking into account the moisture content of the peat. Performing ZTK pretreatment with the hydrodynamic cavitation method, obtaining a homogeneous peat suspension (HP) (Irtiseva et al., 2021, 2022).

The complete technological process of granulation is shown in Fig. 1. The process consists of 3 stages: preparation of raw materials and preparation of a homogeneous mixture, granulation process and drying of granules. Each stage significantly affects the mass proportions of raw materials, moisture content, granule quality, and physical-chemical properties.

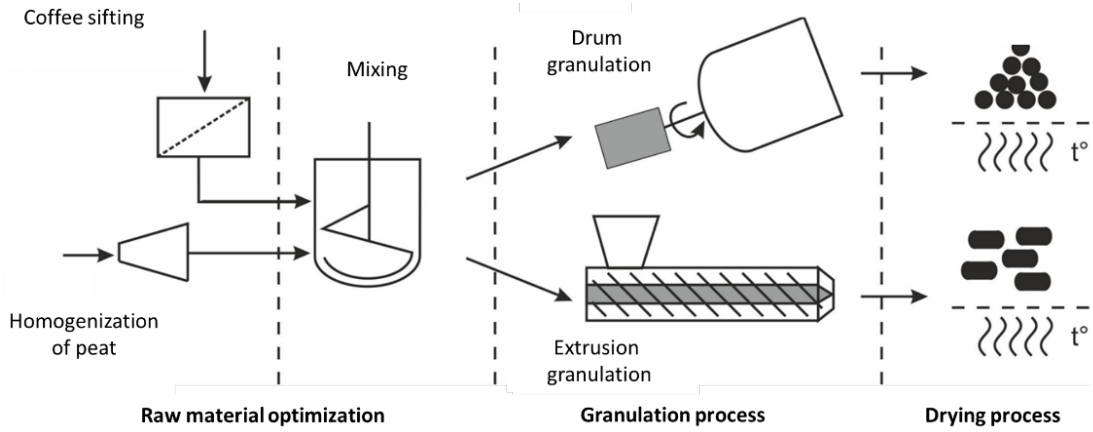

Figure 1. Technological scheme for pellet processing (Vincevica-Gaile et al., 2019).

Fig. 1 shows that 2 granulation methods were used: drum granulation and extrusion. The aim is to identify which granules are mechanically robust and can adsorb more.

Rotary pelletising uses the principle that as the drum containing the mixture rotates, the mixture begins to roll by friction between the mixture and the drum walls, forming agglomerates of particles that further agglomerate to form spherical pellets (Fig. 2). With this technology for producing pellets, the mixture must be precisely prepared so that, as it mixes, the granulation process is initiated. In this process, it was also observed that there was a large excess mass of the mixture, which did not form pellets, which is a significant disadvantage of this technology for producing pellets.

During the experiments, it was observed that the mixture started to granulate badly after a specific time, the reason being the high moisture content of the mixture, which inevitably decreases as it evaporates, so such a mixture needs to be both moistened and

powdered with the dry mixture in order to prevent the granules from dissolving but continuing to roll in the drum. Powdering technology makes it possible to form layered spherical pellets with different compositions.

Since in extrusion pelletisation, the mixture must be drier to form solid pellets (due to the pressure when the mixture is extruded), a smaller amount of binder is used initially than in rotary pelletisation experiments. Similarly to rotational granulation experiments, extrusion granulation experiments vary the composition of the mixture to see the effect of the composition on the resulting granules and their properties.

The pelletising part of the extrusion pelletising machine consists of 2 parts: rollers and a disc with holes. The mixture to be pelletised is introduced into the compartment above these parts. The mixture is discharged under pressure through the slots in the matrix, where a cut-off further adjusts its length. The extrusion pelletising machine used is shown in Fig. 2.

Figure 2. Two machines were used for pelletising: extrusion pelletising: a – working wheel with matrix, b – overall operating diagram of the granulating system. Drum granulation: c – Working rotating cylinder with adjustable angle.

After pelletisation, it is pyrolysed at the following temperatures: 550 °C, 600 °C, and 650 °C. The pyrolysis process converts the carbon to CO_2, thus forming a channel and pores in the pellet. The size of the molecule of the decomposed substance significantly influences the size of the pores.

The source peat is a low-type peat with a 40–50% decomposition rate. SCGs (K), taken from Circle K Latvia, were used after drying to a coffee fraction > 3 mm. Dried peat was used as additional filler.

Moisture analyser KERN MRS 120-3 (UK) is used to determine the moisture content for samples (Table 2). Since the materials used in the work are porous,drying is performed at a temperature of 120 °C, and the mass of the sample is greater than 1 g.

Table 2. Moisture content of components, raw materials

Sample	Moisture, %	Sample	Moisture, %
Raw peat (ZTK)	70.0 ± 2.5	Dry peat (P)	12.0 ± 0.5
Raw SCGs (RK)	67.6 ± 0.6	Peat binder (HP)	94.0 ± 0.5
Dry SCGs (K)	4.0 ± 0.1		

The proportions of raw materials were based on previous experiments (Irtiseva et al., 2022). Initially, experiments were carried out by visual observation of the mixture and its granulation process, from which successful experiments, a specific formulation was further derived, which is further investigated. In the composition tables and for the

raw materials below, the following abbreviations are used: SCGs – K; dried peat – P; peat binder – HP.

The Archimedes method is used to determine the open porosity of bio-based adsorbents: the sample to be studied is completely saturated with a defined liquid, e.g., distilled water (ISO 2738). The resulting biosorbents are subjected to oil sorption testing. The resulting biosorbent is tested in Pilot 10W – 40 SJ/CF semi-synthetic engine oil. The samples were kept together for 15 minutes, with the mass of the oil-impregnated sample being determined every 3, 6, 10 and 15 minutes to observe the adsorption kinetics.

The compressive strength of the specimens was determined using a Moeller (Germany) mechanical strength testing machine.

RESULTS AND DISCUSSION

In order to be able to make a further evaluation of the composition of the final bio-based adsorbent product, the composition of the raw materials needs to be clarified.

Since pyrolysis carbonises organic compounds to form a carbon structure, it is necessary to analyse the mineral composition, as these minerals will be retained in the bio-based adsorbent. The metal content of the raw material used, peat, was determined Summarising the metal concentrations gives the following results: calcium (3384$\mu g \cdot g^{-1}$), aluminium (1,674 $\mu g \cdot g^{-1}$), sulphur (1,538 $\mu g \cdot g^{-1}$), iron (806 $\mu g \cdot g^{-1}$), magnesium (747 $\mu g \cdot g^{-1}$), silicon (436 $\mu g \cdot g^{-1}$), phosphorus (313 $\mu g \cdot g^{-1}$), potassium (747 $\mu g \cdot g^{-1}$), lead (55 $\mu g \cdot g^{-1}$), titan (48 $\mu g \cdot g^{-1}$), manganese (40 $\mu g \cdot g^{-1}$), barium (26 $\mu g \cdot g^{-1}$), sodium (25 $\mu g \cdot g^{-1}$), boron, lithium, strontium, copper, antimony, selenium, arsenic, thallium, vanadium, chromium, cadmium, nickel, cobalt, molybdenum, beryllium have ion concentrations < 10 $\mu g \cdot g^{-1}$.

Table 3 illustrates the recipes suitable for the drum granulation method and extrusion. It should be noted that the granulation methods vary according to the type of technological process, and the recipes also show how the amount of binder mass varies concerning the fillers.

Table 3. Composition of pellets by weight (wt%) and method of pelleting

Composition mass proportion, wt%			
Sample	K	P	HP
Drum Granulation			
1. K-HP	35 ± 1	0	65 ± 1
2. K-HP	45 ± 1	0	55 ± 1
3. K-HP	50 ± 1	0	50 ± 1
4. K-P-HP	12 ± 1	7 ± 1	81 ± 1
5. K-P-HP	14 ± 1	14 ± 1	72 ± 1
Extrusion			
E1	67 ± 1	20 ± 1	13 ± 1
E2	69 ± 1	21 ± 1	10 ± 1
E3	65 ± 1	19 ± 1	16 ± 1
E4	84 ± 1	0	16 ± 1

The mass proportions were based on the principle of a homogeneous mass. Initially, no binder was mixed, to which fillers were added. Each pellet has its mass proportions because of the different methods of pelleting.

Spherical pellets are obtained with a higher binder content, while the extrusion method uses more filler. Table 3 shows that the highest binder content (HP) for spherical pellets is for sample 4. K – P – HP means this sample should have better physical properties and be more durable. The extruded granules show how the highest filler content (K) is for the E4 sample, thus giving good physical properties.

The pyrolysis process retains from the samples pellets that are extruded: E1, E2; E3, E4 and spherical 4. K – P – HP.

The results of the open porosity analysis showed the following: the best result for extruded granules is E2 – 48 ± 4% at 600 °C, and the worst is E4 – 41 ± 3% at 600 °C. However, for spherical granules at 550 °C, 61 ± 4%.

The open porosity data show that the pyrolysis temperatures used have no significant influence on the open porosity of the resulting product. It can be seen that the open porosity of the pellets obtained by rotary pelletisation is higher than that obtained by extrusion pelletisation. The rounded pellets have a significantly lower density than the extruded pellets. These pellets may also have closed pores, which can be converted to open pores by changing the pyrolysis temperature.

The apparent density data show that the rounded pellets are not as compacted in the raw form as the extruded pellets, indicating a higher porosity after pyrolysis treatment. The high errors in the apparent densities of the rounded pellets indicate differences in the composition of the pellets (Fig. 3). It can be concluded that the apparent density may differ from the bulk density by a factor of 1.5 to 2.1. It is important to note that there will be a volume change when the pellets are transported, as the self-weight of the pellets will compact the pellets between them, reducing voids in the buried material and resulting in a denser arrangement.

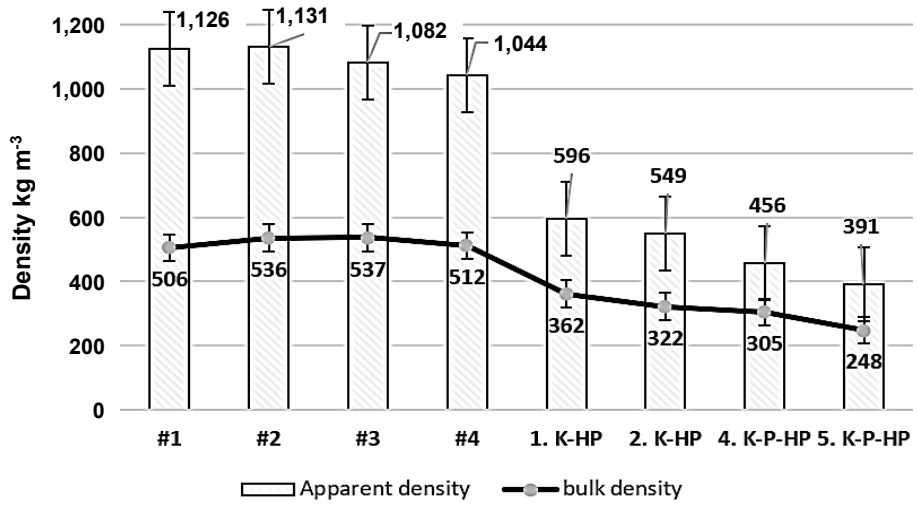

Figure 3. Bulk and apparent densities of the produced pellets before pyrolysis.

The apparent density strength of extruded granules ranges from 900 to 1,100 kg·m^{-3} (Fig. 3). The bulk density is 2 times lower, around 500–540 kg·m^{-3}. Spherical pellets have an apparent friability of 350–650 kg m^{-3}, where the burial density is 1.6 less between 200–400 kg·m^{-3}. The best mechanical properties are found for samples E2-600, E4-550 and E3- 650, which are between 180 and 230 kPa. The mechanical properties of the pyrolysed samples increase by a factor of 1.8 compared to the root sample dried at 105 °C to constant weight (Fig. 4).

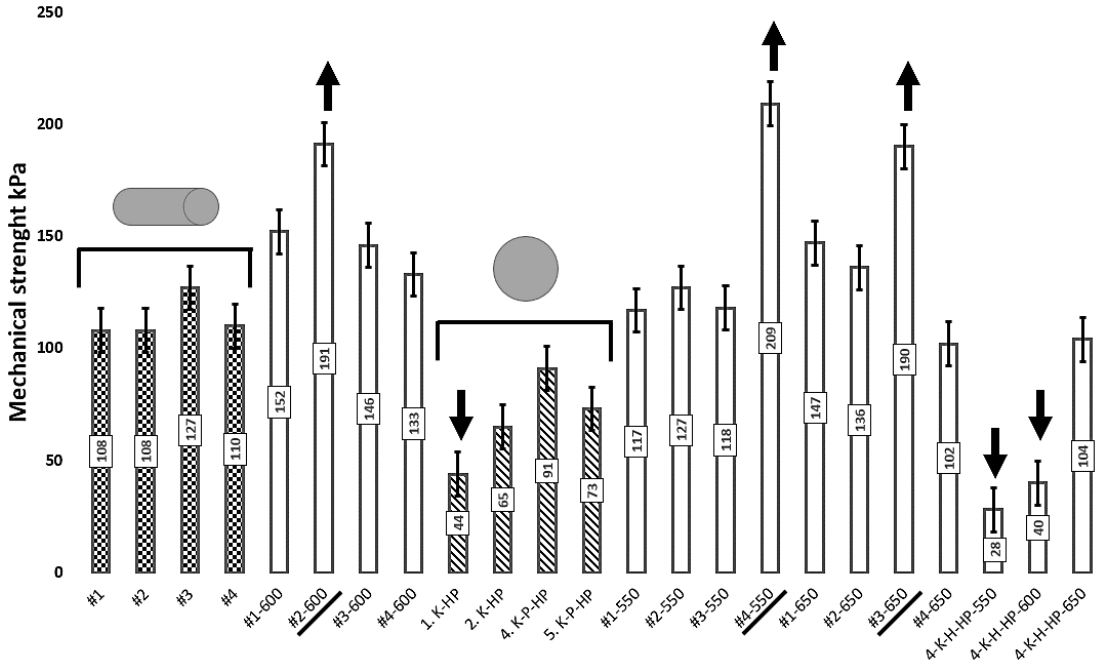

Figure 4. Mechanical properties before and after pyrolysis for extruded and spherical pellets.

The results of the tests on the sorption of the resulting bio-based adsorbents in oil (Pilot 10W – 40 SJ/CF semi-synthetic engine oil) can be seen in the following graphs. In Fig. 5 and Fig. 6, the adsorption capacity is highest at minute 3 for the granules of the E4 – 600 and E4 – 550 compounds, which account for 55–66% of the initial mass.

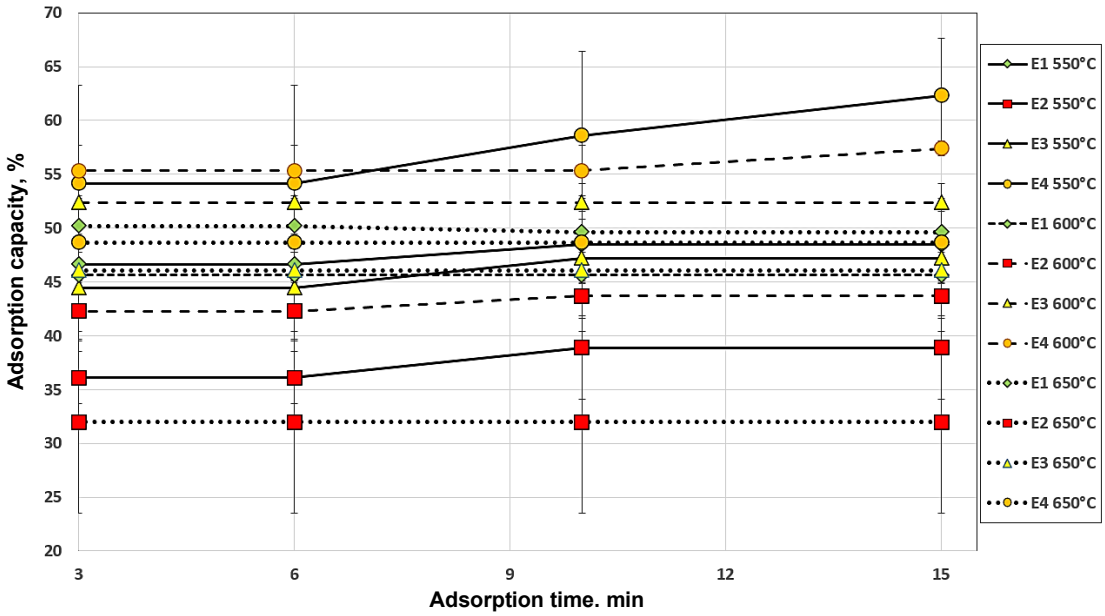

Figure 5. Adsorption kinetics of extruded pellets in Pilot 10W-40 SJ/CF oil.

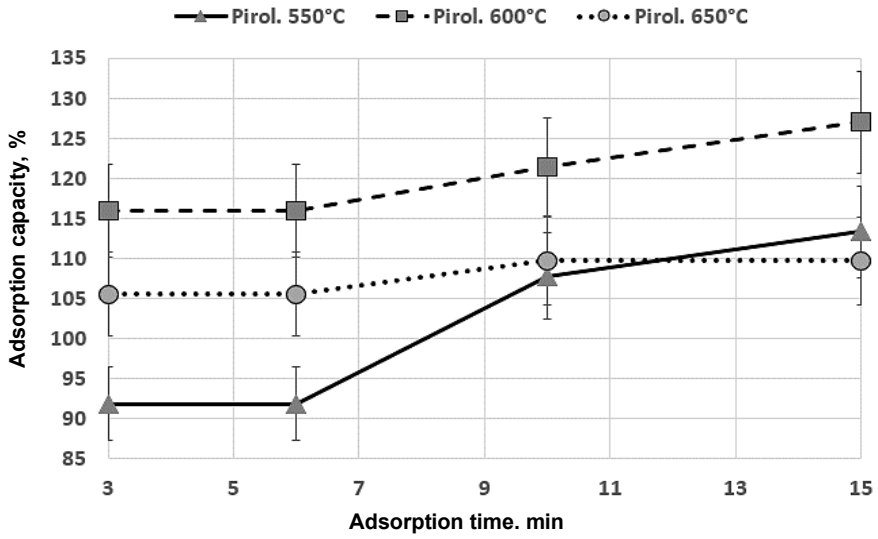

Figure 6. Adsorption kinetics of spheric pellets 4. K – P – HP of Pilot 10W-40 SJ/CF oil.

It can be seen that the bio-based adsorbent produced by rotary granulation adsorbs a greater amount of oil relative to the mass of the sorbent itself. Considering the physical parameters described above, it can be concluded that the resulting rotary granulation bio-based adsorbent has a highly porous structure. The data obtained demonstrate the effectiveness of the bio-based adsorbent in the sorption of petroleum products. When the samples were placed in the oil, air bubbles were observed to be released from the biosorbent within the first few seconds, indicating that sorption was taking place. The same picture can be seen where the same observation is seen in the water. The first observations suggested a reaction of the biosorbent with the water, but no environmental changes were observed when the pH of the water was checked.

Given that the moisture content of the samples is 60–70%, the shelf life of the samples was analysed. For the uncured samples, moulding was observed and the drying process was then carried out (Fig. 7).

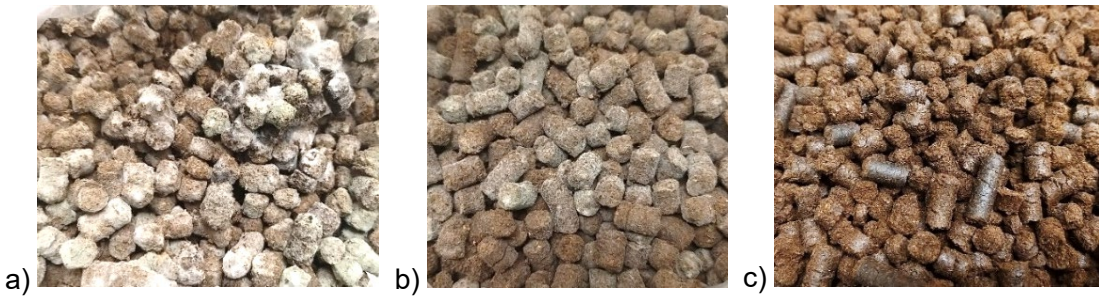

Figure 7. Side effects for samples with and without drying: observation of mould formation in undried, untreated (a), undried with disinfectant treatment (b) and dried untreated (c) pellets after 7 days (a), 14 days (b) 56 days (c).

CONCLUSIONS

This study used SCGs from local Circle K Latvia shops in Riga, Latvia, which were dried at 105 °C until constant weight and sieved. The binder used was ZTK low-type peat, which was subjected to metal analysis and pre-treated with hydrodynamic cavitation to obtain a homogeneous peat suspension. The mass proportions were based on the principle of a homogeneous mass, with spherical pellets having higher binder content and extrusion methods using more filler. The results showed that the best results for extruded granules were E2 – 48 ± 4% at 600 °C, while the worst were E4 – 41 ± 3% at 600 °C. The mechanical properties of pyrolysed samples increased by 1.8 compared to raw samples dried at 105 °C to constant weight. The sorption of the resulting bio-based adsorbents in oil showed the highest adsorption capacity at 3rd minute of the test. E-2 pellets have potential for use in the treatment of soil and water from petroleum products.

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