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Solids removal from Balneo peat extracts

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ABSTRACT

Alkaline extract of mud as well as Balneo peat mud presents antioxidant, anti-inflammatory, astringent, bacteriostatic and bactericidal properties and can be used as cosmetics and pharmaceuticals ingredients. Before application solids present in extract have to be removed. In the present work, two kinds of these processes were considered. A cake filtration process was considered with and without using a filter aid (fine diatomaceous earth). In both cases, the process was unstable in time due to the high resistance caused by the compressible cake formed from the separated solids. Due to the stability in time, a centrifugal sedimentation process was chosen. As optimum conditions to obtain near homogeneous extract (solid content not higher than 0.11 g/L), centrifugation at 6,000 n/min for 2 min was selected. Under this condition, the concentration of matter in the system was about 0.1 g/L.

Keywords: Filtration; Centrifugation; Balneo peat; Extract; Humic acids

1. Introduction

Peat transformation processing involves the accumulation and humification of vegetal debris in long-term conditions and excessive wetting of the topsoil. During humification, the debris is decomposed by the action of various microorganisms and atmospheric agents. The environmental conditions in which peat is formed have a direct influence on the degree of its saturation with minerals, as well as the composition of organic matter. The main constituents of organic matter are organic acids consisting mainly of humic and fulvic acids, salts of the abovementioned organic acids, bitumen, cellulose, lignin and proteins [1, 2].

Humic acids are a humic fraction with very different physicochemical properties [3]. They can be extracted from soil samples using an alkaline solvent extraction. In general, the polymers are composed of a core consisting of aromatic rings, for example, phenols and compounds containing cyclic forms of nitrogen atoms. This core is most often linked to amino acids, sugars, peptides and other aliphatic components. Very important for humic acids are their functional groups. The most commonly encountered groups are carboxyl, phenol, quinone, methoxy, amino, alcohol and carbonyl groups.

Fulvic acids are soil compounds with a broad spectrum of solubilization in water. Comparing with humic acids, they are characterized by lower carbon content and lower molecular weight. Both their aromatic rings and chains are combined with numerous oxygen-rich functional groups [4].

Peat used in natural medicine is called balneological peat, otherwise therapeutic mud. It belongs to peloids, that is, geological works with high organic content and mineral salts. The mulberry contains at least 75% organic compounds in the dry matter and forming the humification degree. The plant remains are usually above 50%.

The weak thermal conductivity of peat and the high thermal capacity are of great importance [5]. Both of these properties guarantee the effect of deep and prolonged warming of the body, which in turn leads to intensive expulsion from the body along with subsequent metabolic products. At the contact of mud skin, these properties provide for the absorption of various toxic substances and their removal from the body.

The biologically active components of mud, mainly humic acids, fulvic acids, sugars, proteins and bituminous

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substances act synergistically to provide anti-inflammatory, astringent, bacteriostatic, and bactericidal properties. A significant number of humic, fulvic and phenolic acids has so-called antioxidant properties. Extracts obtained by the use of sodium base (0.4% m/v) as a solvent in extraction process are rich in these compounds and similarly with antioxidant properties [6, 7]. Humic acids were also effectively extracted by poly(ethylene glycol)-based aqueous biphasic systems [8].

The aim of present research was to select the method and conditions for effective separation of solid residues in extracts of balneological peat. This task is not known in the literature. Only methods for removing water from the soil (peat) before using them as a fuel have been described [9, 10]. Soil (peat) particles are varied in size. They have high ability to swell and flocculation that makes their separation not easily [11, 12]. Due to the applicability of the process on an industrial scale, only processes with the possibility of easy scale transfer were considered.

2. Materials and methods

The tested medium was a fresh alkaline extract obtained after extraction of balneological peat according to the patented technology [13]. Extraction was carried out in a (2 L) stainless steel tank equipped with a mechanical paddle stirrer. Mixing intensity was maintained at 70 rpm. The dimensions of the mixing element (mounted at a height of 6 cm from the bottom of the tank) were 20 mm × 10 mm. 250 g of peat were poured into 1.2 L of 0.4% NaOH solution and the extraction was carried out for 5.5 h at 24°C. After this time, stirring was switched off to allow the suspension to sediment. The solution located above the sediment was collected by a drainage pipe fixed approximately 2 cm above the level of the sediment.

The extract contained solids, estimated on the base of standardized dry mass curve of equation C (g/L) = 1.08 × Abs (550), at 24–29 g/L. Differences in the dry weight in particular samples were taken into account in further calculations. Particles size was analyzed on Laser Diffraction Particle Size Analyzer, SALD-2300 (Shimadzu).

To remove the solids (peat (soil) particles), the extract was alternatively subjected to a sedimentation centrifuging or cake filtration. Each process was tested in duplicate (presented data are the average values).

2.1. Centrifuging

A sedimentation centrifuge from Hettich (Universal 320R) was used in first step of the process analysis. The effects of spin speed and process time were analyzed. The degree of medium heterogeneity was monitored at 550 nm by spectrophotometry. The wavelength of 550 nm was selected based on the spectrum made in the range of 190–1,100 nm on the spectrophotometer Hitachi UV-Vis U-1900. A standardized curve on solids concentration of equation C (g/L) = 1.08 × Abs (550) was used to heterogeneity estimation.

Initially, the process was carried out in a batch sediment centrifuge (Universal 320R, Hettich). The tested parameters were: time (in range of 1–10 min) and spin speed (in range of 500–6,000 n/min). Selected results obtained in batch processes at given parameters were verified in a continuous centrifuge delivered by Pennwalt Limited (model AS-12) (Fig. 1).

2.2. Cake filtration

The filtration process was carried out using a laboratory set supplied by ChemTech (Fig. 2). The displacement tank had a capacity of 1.5 L and a suspension tank of 2.2 L. Their material was stainless steel 1.4301/1.4307. A flat nylon filter

Fig. 1. A continuous centrifuge from Pennwalt Limited (USA, model AS-12).

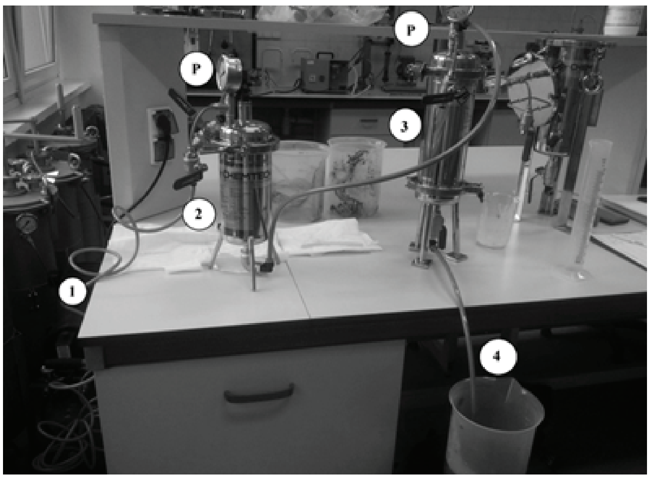

Fig. 2. Research station for overpressure filtration (1, air compressor; 2, displacement tank; 3, tank containing filter; 4, filtrate tank; and P, pressure gauge).

(ChemTech, Poland) with 50 μm pores and a filtration area 78.5 cm^2 was used.

The substance used as a filter aid was fine diatomaceous earth (Celite Standard Supercel, Aces, Poland). The solution with diatomaceous earth was filtrated before main process at 0.08 MPa. It was used in the amount of 10 or 20 g on the filter surface (a surface concentration was 0.127 and 0.255 g/cm^2, respectively). The obtained cake was then rinsed twice with water.

During main filtration overpressure was maintained at 0.06 or 0.08 MPa. Filtrates were checked in the aspect of their heterogeneity by measurements at 550 nm and a standard curve C (g/L) = 1.08 × Abs (550) was used.

3. Results and their discussion

3.1. Centrifuging

Centrifuging process was tested initially in a batch sediment centrifuge. Table 1 shows the obtained results. The process efficiency at different process duration and at different spin rate was expressed by solids concentration in supernatant.

Centrifuging process was very efficient. Even at low values of parameters (1 min, 500 n/min) only 5% of solids remained in the solution. These results are satisfactory because gravitational sedimentation of soil and peat is very slow [14, 15].

As can be seen in Table 1 the higher spin speed, the higher solution clarity. The speed 6,000 n/min was the highest possible value for Hettich (Universal 320R). Extend time from 2 up to 10 min at spin speed above 2,000 n/min did not bring significant benefits (Fig. 3). Hence, a short centrifugation time was selected at high revolutions of the centrifuge (6,000 rpm, time 2 min). Under the indicated conditions, the concentration of matter in the system was about 0.1 g/L. To removing remaining particles, a polymeric membrane should be the best choice [16]. The extract rich in humic acids can be directed subsequently to capillary isoelectric focusing [17].

The spinning time of 2 min at 6,000 n/min was verified positively (relative error 4.2%) in a continuous centrifuge from Pennwalt Limited (USA, model AS-12).

3.2. Cake filtration

During the centrifugation process, the extract used during cake filtration contains solid at concentration of 24–29 g/L.

Table 1
Solids concentrations (g/L) after centrifugation at given time and spin speed

Spin speed (n/min)	Time (min)				
	1	2	4	7	10
500	1.213	0.778	0.582	0.443	0.400
1,000	0.867	0.540	0.412	0.389	0.378
2,000	0.612	0.385	0.362	0.346	0.335
4,000	0.428	0.227	0.211	0.199	0.194
6,000	0.265	0.108[a]	0.108	0.108	0.108

Note: The initial solids concentration was in the range of 24–29 g/L.
[a]Corresponds to the optimal conditions.

Filtration was carried out at constant value of overpressure (0.06 or 0.08 MPa) and with different amount of diatomaceous earth (0, 10 and 20 g). The size of the filter pores equal 50 μm was selected after particles size analysis made by the use of laser diffraction particle size analyzer. The range of their diameter in extract at concentration in range 24–29 g/L was quite high (75–540 μm) because the particles were sticking to one another.

A strong decrease of the filtration stream similar for both pressures (Fig. 4) was observed. The initial stream was strongly dependent on the aid mass (Fig. 5), which means that resistance coming from the cake of diatomaceous earth was not to neglect.

The cake formed by solids from peat was highly compressible, which caused filtration, the most common method to heterogeneity removal [18, 19] has failed.

The filter aid (fine diatomaceous earth) affected the quality of the filtrate only in the first minutes of the process (Fig. 6). After approximately 20 min, the filter cake formed from the retained particles of peat had a dominant influence on the filtration efficiency. Therefore, in the literature there are many applications of peat used to cake forming during various media clarification [20–22].

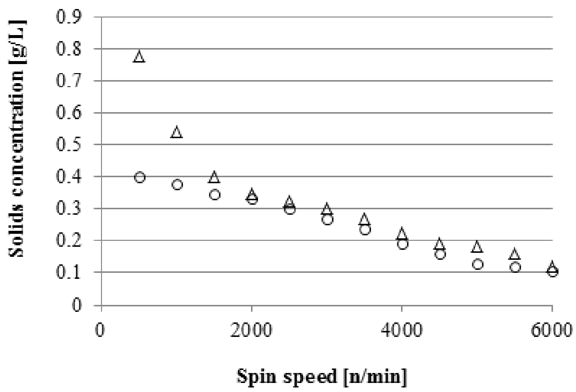

Fig. 3. Effect of spin speed and time: 2 (Δ), 10 (o) minutes of centrifugation on the system heterogeneity expressed by solid concentration in supernatants. Initial concentration was in the range of 24–29 g/L.

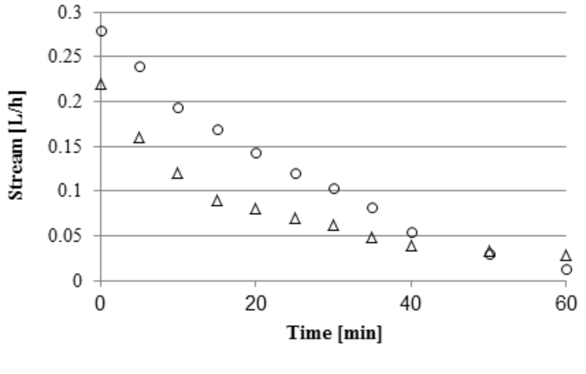

Fig. 4. Change of filtrate flow in time with overpressure of 0.06 (Δ) and 0.08 (o) MPa (filtration area 78.5 cm^2, initial solids concentration approximately 26 g/L, amount of filter aid 20 g).

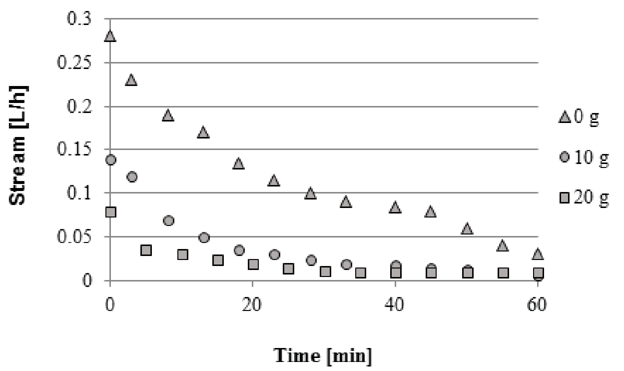

Fig. 5. Change of stream over time depending on the amount of diatomaceous earth (filtration area 78.5 cm^2, 0.08 MPa, initial solids concentration approximately 26 g/L).

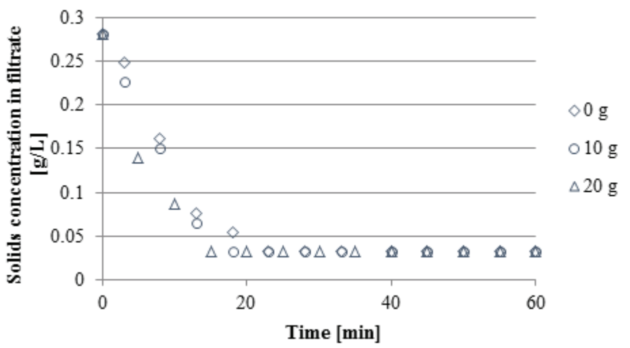

Fig. 6. Quality of filtrate expressed by the solids concentration as a function of time and amount of filter aid per 78.5 cm^2 of filter surface (0.08 MPa, initial solids concentration 26 g/L).

The clarity of filtrates was better than ones in supernatants from centrifugal sedimentation. The concentration of solids was approximately 0.03 and 0.11 g/L in filtrates and supernatants, respectively. However, the sharp decrease filtrate stream (Figs. 4 and 5) indicates that one filtration cycle would not be so long. Increasing resistances in filtration process coming from the changes in peat particles structure located on a filter surface [23].

4. Conclusion

The process of centrifugal sedimentation was indicated to purify the peat extract from the solid residues. The stability of this process should not be affected by the variability in a crude extract quality (that cannot be said about cake filtration). As optimum conditions to obtain near homogeneous extract (solid content lower than 0.11 g/L), centrifugation at 6,000 n/min for 2 min was recommended. A continuous centrifugation under these conditions also gives positive results.

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