UNIVERSITY OF LATVIA
FACULTY OF GEOGRAPHY AND EARTH SCIENCES
DEPARTMENT OF ENVIRONMENTAL SCIENCE

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SAPROPEL FOR THE DEVELOPMENT OF BIOCOMPOSITE MATERIALS: PROPERTIES AND APPLICATION POSSIBILITIES

DOCTORAL THESIS
Submitted for the PhD Degree in Earth Sciences, Physical Geography and Environmental Sciences
Subfield – Environmental Sciences

PROMOCIJAS DARBS
Zinātņu doktores grāda iegūšanai zemes zinātnēs, fiziskajā ģeogrāfijā, vides zinātnēs

Riga, 2021

The research for Doctoral Thesis was carried out at the University of Latvia, Faculty of Geography and Earth Sciences, Department of Environmental Science,

over the period from 2015 to 2021.

NATIONAL DEVELOPMENT PLAN 2020 | EUROPEAN UNION European Social Fund

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This work has been supported by:

ESF Project Nr.8.2.2.0/18/A/010 "Academic Staff Renewal and Continuing Proffessional Development at the University of Latvia"
Latvian Council of Science for providing the grant 'Properties and structure of peat humic substances and possibilities of their modification'
No. lzp-2018/1-0009

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The thesis will be defended at the public online session of the Doctoral Committee of Environmental Science, the University of Latvia, at the Faculty of Geography and Earth Sciences), at 10:00 on August 26, 2021.

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Abstract

The aim of the doctoral thesis "SAPROPEL FOR THE DEVELOPMENT OF BIOCOMPOSITE MATERIALS: PROPERTIES AND APPLICATION POSSIBILITIES" is to study properties of sapropel and possibilities to use it for development of biocomposites for applications in agriculture, construction industries and other fields as well as to test properties of obtained materials in respect to their application possibilities. Samples of biocomposite materials were created by using different types of sapropel as binder and birch wood grinding dust, birch wood fibre, hemp shives and fibre aerosil, mahagony sawdust as fillers. Birch wood veneer and beech wood planks, peat was used for testing sapropel adhesive properties. In the theoretical part of thesis is offered an overview about sapropel, possibilities of utilization, characteristics and possibilities to use for design of environmentally friendly construction materials. The created composite materials were analyzed at the results section. Measuring of mechanical strength, thermal conductivity, microbial stability, biodegradation, ageing of composite materials, compressive and flexural strength of composite materials, sound insulation properties, comparison of auto-ignition were carried out and analyzed. Thesis convincingly demonstrates that using local resources such as sapropel and by-products of the production process, such as birch wood sanding dust, birch wood fibers, hemp shives and wood chips, it is possible to develop environmentally friendly composite materials in construction and agriculture, adapting them to the needs of use. Biological stability of natural sapropel containing biocomposites is one of key parameters for their application potential and should include detailed evaluation of composites in respect to major groups of microorganisms of concern. The mechanical and thermal properties of sapropel-based composites were similar to those of synthetic as well as mineral materials, suggesting that sapropel composites could have similar use in the construction industry.

The thesis summary consists of 28 pages with 1 figure and 4 tables.

Key words: sapropel, binder, composite material, microbial stability, biodegradation

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Introduction

The contemporary economy worldwide is based mainly on fossil material use (Ingrao et al., 2018), and this approach significantly contributes to the depletion of resources and environmental problems, especially climate change. Bioeconomy (biotechonomy) can be considered as an alternative to fossil material-based economy. It implies using biomass or biotechnology to produce goods, services or energy (Lewandowski, 2018). Taking into account global environmental and climate problems as well as the decrease and depletion of global resources, a need to reduce the use of synthetic chemicals and to develop a bioeconomy is necessary. Expanded use of bio-based materials can be considered as an action tool within the sustainable development strategies in European Union (Altozano, 2012) and in Latvia (Latvijas Republikas Zemkopības Ministrija, 2017). Thus, it is crucial to search for applications of natural materials as a replacement of synthetic ones (Fava et al., 2015).

Organic-rich lake sediments such as sapropel, gyttja are believed to be prospective materials for diverse applications (Balčiūnas et al., 2016; Stankevica, 2020). Sapropel is regarded as a waste product of lake reclamation (an action actual for eutrophic lakes) and could be required to remove sediment bulk mass to maintain the lake ecosystem by not allowing overgrowth. Thus, sapropel processing and use are sustainable from the perspective of circular economy with the additional benefit of developing sapropel-based products required in the market. In Latvia, the majority of lakes are eutrophic, with significant impacts of anthropogenic eutrophication and the total volume of sapropel resources is ~2 billion m^3. Sapropel reserves in Latvia's lakes reach 700 - 800 million m^3, while sapropel reserves in bogs reach 1.5 billion m^3 (Segliņš, 2014). Sapropel can be considered a renewable resource since lake eutrophication, being a natural process, is continuous. Thus, the development of new possibilities to facilitate sapropel application can support the reclamation of lakes, as well as can significantly contribute to the development of bioeconomy.

Sapropel properties limit its direct application that is usually tended to agricultural applications. Together with a limited level of research and often outdated application studies, it results in a lack of interest from industries involved in the development of new products. One of the remarkable properties of sapropel is its ability upon processing and drying to act as adhesive and/or binder for other materials (Balčiūnas et al., 2016; Gružāns, 1958, 1960; Klavins and Obuka, 2018). Thus, sapropel can be used to develop composites based on the combination of properties of different groups of materials and to develop new materials with new characteristics. New composite materials is a general trend of material development nowadays. Commonly, composites are synthetic material based, e.g. mineral wool, stone wool, glass wool, while unique properties of sapropel support allow introducing a new group of composite materials formed by a matrix and a reinforcement of fibers - biocomposites (Mohanty et al., 2000). Nevertheless, sapropel has not been used for the production of biocomposites, but this direction of research can is believed to be highly prospective since natural material-based binders for biocomposites are limited.

One of the main challenges of working with sapropel is that its properties limit its direct application to the development of sapropel based composites includes options to use waste biomass as fillers. Such approach provides possibilities to find new applications of waste materials, such as hemp shives, wood sanding dust and wood chips. Another challenge in developing biocomposites is proper testing of their properties (Bulota et al., 2011; Jawaid et al., 2019; Mngomezulu et al., 2014). Biocomposite testing traditionally concentrated on testing functional properties (mechanical strength, durability of use etc.), but the biological stability tests often are neglected as the testing methodologies are not elaborated, and their functioning has not been much studied. However, as biocomposites might be subjected to biological degradation, the biological stability studies are of utmost significance for estimation of their more comprehensive application.

The aim of the doctoral thesis is to study properties of sapropel and possibilities to use it for development of biocomposites for applications in agriculture, construction industries and other fields as well as to test properties of obtained materials in respect to their application possibilities.

The worktasks of the thesis includes:

1. Study of sapropel properties, relevant for development of biocomposite materials
2. Develop principles for biocomposite material production using sapropel
3. Prepare new, application oriented sapropel based biocomposites
4. Develop biocomposite material testing methods as well as analyse and evaluate sapropel composite material application possibilities

Hypothesis

Elaboration and aprobation of new testing methodologies are essential, to develop application oriented, sapropel based biocomposites, which includes detailed studies of elaborated material biostability in addition to functional property estimation.

Scientific and applied significance

1. Development of new approach for lake recultivation waste product – sapropel use for production of natural material based biocomposites and demonstration of the approach efficiency
2. Creation of design concepts of biomaterial based biocomposite material using sapropel
3. Preparation of sapropel based biocomposites for new, market oriented applications in building material industry and design, as well as in other fields, thus supporting use of local, natural based material use
4. Elaboration of sapropel based biocomposite analytical characterization and testing methodology, to prove their biostability, functional properties and application potential

Approbation of the results

The results of the doctoral Thesis are published in 9 scientific articles, including 8 articles in SCOPUS; Web of Science and discussed in 5 international as well 6 local scientific conferences.

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1. Literature review

1.1. Sapropel: formation conditions and composition

Sapropel is a partially renewable resource (Segliņš and Brangulis, 1996) formed under differing conditions. Complex formation conditions reflects problems of terminology as recently is discussed in thesis work (Stankevica, 2020). Sapropel is a loose and fine-grained sediment rich in organic matter in inland waters (Emeis, 2009). Sapropel has a low content of inorganic biogenic components and a mixture of mineral nature (Stankeviča and Kļaviņš, 2014). Sapropel organic matter mainly consists of residues of aquatic plants and aquatic animals - phytoplankton and zooplankton (Kurzo et al., 2004), which reproduce in large quantities in stagnant or poorly flowing overgrown water basins (Lācis, 2003). Sapropel consists of diatoms, green algae and cyanobacteria, as well as foraminifera, radiolaria, dinoflagellates, various species of crustaceans, sponges and bacteria.

There are two types of sapropel deposits: terrestrial, which are located in bogs under peat layer and in lakes (under water) (Kaķītis, 1999). According to the results of Latvian lakes research, sapropel reserves in Latvia's lakes reach 700 - 800 million m^3, while sapropel reserves in bogs reach 1.5 billion m^3. Total sapropel resources in Latvia can be estimated at 2 billion m^3 (Segliņš, 2014) however the estimation of the accumulated resources should be re-evaluated considering results of recent studies (Stankevica, 2020).

One of the most important properties of moist sapropel is its colloidal structure, which determines the ability of the organic colloidal particles of sapropel to absorb large amounts of water, resulting in a high moisture capacity of 70-97% (Vimba, 1956) and low filtration rate (Liužinas et al., 2005). The relative humidity of sapropel is related to its organic content - the higher the organic content, the higher the moisture content (Stankevica, 2020).

1.2. Sapropel application possibilities

Sapropel can be found in significant quantities and thus of importance is to study its application possibilities. Sapropel can be considered as a renewable and natural material and for example, in case of lake recultivation, involving removal of sediments it can be considered as a waste material and thus available for use as low-cost product. Sapropel, like peat, has a wide range of possible uses, which vary with the composition, properties and availability of the sapropel resource. It can be used in various sectors of the economy, such as agriculture, medicine, veterinary, construction industry, etc.

From the point of view of use, organic sapropel is accepted as the most valuable type of sapropel, however, this does not mean that this type is versatile and produces equally good results in all areas of use of sapropel (Brakšs et al., 1960; Gružāns, 1960; Штин, 2005).

Agricultural applications of sapropel have been most widely studied. Sapropel properties influence its application potential in agriculture: 1) sapropel can support development and optimisation of soil structure, 2) sapropel can enrich soil with organic matter, especially with humic substances, 3) sapropel is a source of nutrients and can ensure slow release of nutrients 4) biologically active substances in sapropel can support plant growth (sapropel can act as biostimulant). In Latvia, the use of sapropel in field fertilization has been studied at the Latvian Academy of Agriculture since 1954. As an organic fertilizer, sapropel can be used both fresh and as a compost or substrate. It is important to note that freshly obtained, non-aerated sapropel contains zinc, aluminum, non-oxidized iron compounds and bituminous substances that may inhibit its conversion into plant nutrients during the first year of use and reduce soil microorganism activity (Kaķītis, 1999). Although the sapropel is highly effective in the first year of application, it is mixed with manure or sludge.

Considering properties of sapropel, there is good potential to develop sapropel applications in animal husbandry to support the efficiency of livestock farming, the efficient use of bird and animal feed. Argument in respect to use of sapropel are related to its biogenic nature and evident need to reduce use of synthetic products.

One of the uses of sapropel is a dietary supplement in animal husbandry that would increase the live weight of animals without the use of chemical preparations. Organic sapropel, which is natural and safe for poultry, is usually used for this purpose. When enriched with natural vitamins and micronutrients, sapropel can be used in animal husbandry (livestock farming) as a vitamin and micronutrient supplement.

Sapropel aplication potential in healthcare is backed up with a long history of balneology based on use of peloids or medical mud for external applications and baths as well as other preparations and materials. As peloids are described organic-mineral complexes with high concentrations of organic matter and others that are used in therapeutic procedures (Badalov and Krikorova, 2012). Main factors affecting efficiency of sapropel use in balneology includes known presence of numerous biologically active substances such as hormones, sterols, amino acids and vitamins (Szajdak and Maryganova, 2007). Sapropel is used in balneotherapy and cosmetology (Badalov and Krikorova, 2012) as extracted from their source (lake or bog) and in combination with physical factors (sapropel moisture, heat holding capacity) psychological aspects (relaxing atmosphere) its application convincingly demonstrates biostimulating effects, activate metabolism and immune system (Anderson, 1996). Sapropel applications in several studies have demonstrated high efficiency to treat bone and muscle disorders, joint and spine diseases, myositis, ulcers as well as positive impact on nervous system disorders (Bellometti et al., 1996, 2000). Of importance is the sapropel capacity to reduce inflammatory processes as well as skin diseases especially chronic eczema and several forms of dermatitis (Carabelli et al., 1998).

Organic and mineral compounds in sapropel determine its effectiveness in treating a variety of diseases such as musculoskeletal disorders, peripheral and nervous system diseases, gastrointestinal disorders. Studies on the metabolic activity of sapropel have shown that the healing properties of sapropel are determined by its organic components, including humic substances (Курзо, 2005). Sapropel is used in the treatment of chronic gastritis, mastitis, duodenal and ulcer diseases, furunculus, skin (dermatitis, eczema, and burns) as well as in the treatment of hepatobiliary disease (Штин, 2005).

One of the alternatives to the use of sapropel is its use in the production of briquettes and pellets for heating residential and household buildings (Kozlovska-Kędziora and Petraitis, 2011; J Kozlovska and Petraitis, 2012; Курзо, 2005; Штин, 2005). The use of sapropel briquettes saves other energy resources because the burning process of sapropel briquettes is longer than that of conventional briquettes (Kozlovska-Kędziora and Petraitis, 2011; Justyna Kozlovska, 2012). In the production of these briquettes, sapropel can be mixed with straw, sawdust or peat (Штин, 2005). Studies have shown that the concentration of contamination during the burning process of sapropel briquettes does not exceed regulatory limits. Briquettes of this type can reduce pollution and ensure a smooth use of the energy source (Justyna Kozlovska, 2012). The use of sawdust-sapropel briquettes results in lower CO_2 emissions and improved combustion (Штин, 2005).

A lot of research has been done into the use of sapropel in the chemical industry. Due to its chemical composition, availability, relatively low cost and ecological safety, sapropel is one of the most suitable raw materials for drilling solutions, which reduce friction and are required for geological exploration wells (Штин, 2005). Sapropel contains high molecular weight substances: humic substances, natural biopolymers, cellulose, carbohydrates, lignin, hemicellulose and bitumens, its solution dispersion rheological properties and inhibitory activity properties on metallic surface corrosion determine the use of sapropel working fluids in drilling plant hydro systems, and diatomaceous dispersions (Курзо, 2005; Штин, 2005).

1.3. Use of sapropel for development of biocomposites

For many applications growing attention gain composite materials and especially biocomposites. Biocomposites are composite materials formed by a matrix (resin, glue etc) and a reinforcement of fibrous material. Environmental concern and cost of synthetic fibres have led the foundation of using natural fibre as reinforcement in polymeric or mineral composites where the matrix phase is formed by renewable or nonrenewable resources. Composite materials find new and exciting applications in different areas, but the dominant ones are building and construction material industries.

The use of sapropel in the building industry for production of construction materials have been studied.

There are studies about technical tests of sapropel concrete (Brakšs et al., 1960; Gružāns, 1960), sapropel - hemp shives (Pleiksnis et al., 2016; Pleikšnis and Dovgiallo, 2015) and sapropel - wood chips (Obuka et al., 2014) composites. One of the latest study has been done by G. Balčiunas, who have studied sapropel-hemp-paper production waste (PPW) composite materials properties (Balčiūnas et al., 2016). In these studies, the researchers concluded that the use of sapropel as a binder with different materials is such that technical quality can be included in the category of thermal insulation materials for finished products. According to the literature, sapropel can be used as binder for various wood waste, unused waste from the paper and cardboard industry, flax processing (Курзо, 2005), degraded peat and similar raw materials (Gružāns, 1958, 1960) containing composites. Sapropel is a good substitute for protein-based glues, for example, albumin and possibilities to replace proteins would be a significant benefit of sapropel application.

Sapropel with an organic content of more than 85% and nitrogen more than 3.3% can be used for the production of sapropel binder (Курзо, 2005).

The adhesive properties of sapropel can be used in the production of building materials, both cold-pressed and hot-pressed composites are stable at elevated temperatures and pressures (Brakšs and Miļins, 1960).

One of the properties of sapropel is its ability to bind large amounts of water. When producing thermal insulation materials, it is important to keep the material to a minimum shrinkage. Inventors and researchers of sapropel concrete recommend the use of sapropel with moisture up to 60% and filler materials with moisture below 20% to reduce shrinkage.

One of the most important tasks producing the building materials in the future, is to lower energy use at all stages of their lifecycles, from construction to end of use (Asdrubali et al., 2015).

Materials and methods

2.1. Sapropel samples used in the study

In the thesis organic rich freshwater sapropel were used. Sapropel sediments were sampled from four lakes in Latvia – Padelis, Pilvelu, Veveru located in Rezekne district, Latgale region. Piksteres lake - located in Jekabpils District, Selonia Region, Latvia.

2.2. Characterisation of sapropel samples

Loss on ignition. To estimate moisture, carbonate matter content and organic matter of sediments loss on ignition (LOI) method was used (Heiri et al., 1993).

Biological composition. The biological composition of samples was determined with a light microscope, counting and expressing as a percentage of organic matter content by groups in all identified groups of organic residues. Sapropel type, class, grade and application possibilities were identified using sapropel type classification (Stankeviča and Kļaviņš, 2014).

2.3. Materials used for the development of biocomposites and their characterization

Hemp shives, birch wood sanding dust (wood dust) and fiber (wood fibers) were selected as fillers for production of composite materials. In producing of composites, an additional thickening additive filler was used - colloidal silica product "Aerosil". The wider description of the materials can be found in the article – (Obuka et al., 2015).

Block peat (Ltd "Laflora") was also used for composite materials biodegradation studies as a control material. The wider description of the materials can be found in the article - (Obuka et al., 2019).

Birch wood veneer was used for the preparation of plywood. Samples for determination of tensile shear strength: beech wood planks were used. Peat samples: dried natural peat was used for tests as well. The wider description of the materials can be found in the article - (Obuka et al., 2016)

To develop sapropel, peat, wood chip thermal insulation boards - Baložu peat field peat, Pilvelu lake sapropel and wood chips were used. The wider description of the materials can be found in the article - (Obuka et al., 2014)

In addition to sapropel also commercially available binders were used: magnesium oxychloride cement (MOC), hydraulic lime (HL), formulated hydraulic lime (FHL), magnesium phosphate cement (MPC). The wider description of the binders can be found in the article - (Obuka et al., 2017)

2.4. Composite material preparation and curing

To develop composite materials for microbial stability tests as a binder (adhesive) raw sapropel was used. For reference in some cases also inorganic binders were added. Additional information about composite materials preparation and curing can be found in article - (Obuka et al., 2017).

For the composite material preparation and curing for tests, where sapropel was tested as an adhesive, the sapropel samples were mixed completely just before the preparation of three-layer plywood of dimensions 4×250×250 mm. Additional information about composite materials preparation and curing can be found in article - (Obuka et al., 2016).

For the preparation of sapropel, peat, wood chip thermal insulation boards, the activated peat mass with binder properties was obtained by mechanical treatment of peat in the thermal bullet planetary mill RETSCH PM 400. Additional information about composite materials preparation and curing can be found in article - (Obuka et al., 2014)

2.5. Biocomposite material testing methods

ALINA LIFE™ organoclay coating was added to materials in 4% concentration of dry mass (wood fibre or dust in SWD (sapropel – wood dust), SWF (sapropel wood fibre). Additional information about sample preparation for microbial stability tests is found in article - (Obuka et al., 2017). Ageing of obtained composite materials in climate chamber was done by exposing the samples to 30 freeze-thaw cycles (Obuka et al., 2017).

The thermal conductivity was measured using LaserComp FOX 600 heat-flow measurer (Obuka et al., 2015). For testing compressive and flexural strength, the samples were specially prepared (sawed in necessary dimensions). Additional information about this method is found in article - (Obuka et al., 2015).

For the composite material mechanical strength tests (static bending strength, shear strength test, tensile strength), where sapropel was tested as an adhesive additional information about method is found in article - (Obuka et al., 2016).

The materials were also tested for sound insulation. Additional information about method is found in article - (Obuka et al., 2014).

Simple combustion tests were performed to judge the combustion characteristics of the composite materials (Obuka et al., 2014).

In order to compare the potential biodegradability of the composite materials tested, an experimental scheme was developed (Obuka et al., 2019). The microbial respiration was tested according to (Rowell, 2014; Zibilske, 1994; Гавиленко et al., 1975) with some modifications (Obuka et al., 2019).

The respiration assay used in this study, was attributed to the substrate induced respiration (SIR), because carbon sources were added to the soil with specimen. However, the standard principles of SIR measurement (e.g., incubation for 4h) were not considered because of specific tasks of this study. In particular, bioaugmentation and a 7 day incubation was performed for stimulation of the biodegradation process.

After 7-day incubation period the fluorescein diacetate (FDA) hydrolytic activity of microorganisms was tested (Obuka et al., 2019).

Together 3 experiments were done in microbial stability tests. Comparison of microbiological resistance of sapropel-based composite materials, LHC and MHC was carried out. Artificial infection with fungi *Alternaria alternata* MSCL 280 and *Cladosporium herbarum* MSCL 258 was used in microbiological stability tests. Additional information about method is in article - (Obuka et al., 2017).

Two experiments were conducted to determine the microbiological stability in test part two. In both experiment stages, material samples were artificially inoculated with six fungal strains:

1. *Aspergillus versicolor* MSCL 1346;
2. *Penicillium chrysogenum* MSCL 281;
3. *Alternaria alternata* MSCL 280;
4. *Cladosporium herbarum* MSCL 258;
5. *Chaetomium* sp. MSCL 851;
6. *Trichoderma asperellum* MSCL 309.

Additional information about method is in article - (Obuka et al., 2021).

In the first stage of the experiment, the analysed material samples were incubated in two humidity modes - RH 75% and 99% - and at 20 °C (Obuka et al., 2021).

In the second stage of the experiment, samples were kept only at relative humidity 99% and temperature 20±2 °C (Obuka et al., 2021).

For the first stage of the experiment in part two - ACTICIDE FD, were used for additional biological protection tests. In the second stage of the experiment, the biocide BACTERICIDE. In the both the experiments, ALINA *Ltd.* product ALINA LIFE^{TM} organoclay coating was used for additional biological protection tests (Obuka et al., 2021).

Sapropel, peat granules for the agricultural purposes were tested to determine the physical-mechanical properties of sapropel granules for 3 types of granules (pure sapropel, peaty sapropel, sapropel-peat granules). Burial density was determined using the standard LVS EN 1097-3 (Anonymous, 1999), water absorption using the standard LVS EN 1097-6 (Anonymous, 2013), testing and pellet compressive strength testing using the standard EN 1606 (Anonymous, 2007), environmental acidity reaction and electrical conductivity.

To prepare biochar-sapropel granules for agriculture as filler materials were used biochar. Two kinds of biochar were used: biochar (B), deciduous tree biochar (LB.)

Granules composite materials were created by manually mixing wet sapropel and biochar until homogenous consistency was reached. Different ratios of biochar and sapropel were used (1:3, 1:4, 1:6, 1:8 and 1:10) to determine best option. The mass was further divided in two samples and each part was put in prepared metal forms. One sample was air dried (relative air humidity 14-20%), while the other was oven dried in 80 °C temperature. Air dried sample was weighted every hour for first 3 hours and once more after 24 hours of creation moment. Oven dried sample was weighted every 15 minutes. Biochar – sapropel granules granulation, specific surface area, water absorption and mechanical strength tests as described in article - (Vincevica-Gaile et al., 2019).

3. Results and discussion

3.1. Sapropel properties

Sapropel sediments were sampled from four lakes in Latvia – Padelis, Pilvelu, Veveru located in Rezekne district, Latgale region. Piksteres lake is located in Jekabpils District, Latgale Region, Latvia. The sapropel from these lakes has been studied previously and can be considered as prospective for development of composite materials. Characteristics of the sapropel samples are listed in Table 3.1.

Table 3.1. Characteristics of the sapropel samples

Lake	Moisture, %	Organic matter, %	Carbonates, %	Density, g/cm^3
Padelis	85.97	15.27	35.57	1.24
Pilvelu	94.99	84.51	1.26	1.10
Veveru	97.66	86.25	1.18	1.08
Piksteres	96.45	82.67	17.33	1.028

Used sapropel samples represent sapropel types prospective for use as binding materials.

3.2. Development of sapropel composite materials

As prospective components to design sapropel based composites can be considered natural fibers and at first – hemp. To reveal potential of sapropel-lime binder in hemp concrete composites it has been suggested to use sapropel-lime, magnesium oxide-chloride, and lime binders. The obtained compositions were compared with each other and with data available in the literature. Hemp shives, wood fiber and birch wood sanding dust have been used as filler for composite materials as well. These fillers are agricultural and wood processing industry by-products that need to find reusability.

The aim of the study was to find out the potential of composite materials using sapropel and lime as binder and hemp shives, wood fiber and birch wood as a filler and to determine their optimum properties. Density, thermal conductivity, and compressive strength were determined for the specimens produced. The compressive strength of the various composite materials in the tests is shown in the Table 3.2, and it shows that the obtained materials can be used as thermal insulation materials, because their strength meets the requirements of the regulatory framework.

Samples were prepared using mechanical mixing until homogeneous composition were obtained and the sequence of the addition of components were mixed.

Table 3.2. Characteristics of composite material

<table>
<tr><th>Designation</th><th>Composition</th><th colspan="2">Characteristics</th></tr>
<tr><td rowspan="4">Sapropel-lime-hemp composite SLHC 1-2</td><td rowspan="4">Sapropel
Lime
Hemp shives</td><td>Raw material ratio (filler:binder:binder)</td><td>1:2.5:2.5</td></tr>
<tr><td>Density kg/m^3</td><td>306.88;296.31</td></tr>
<tr><td>Thermal conductivity, W/m·K</td><td>-</td></tr>
<tr><td>Mechanical strenght, MPa</td><td>0.25</td></tr>
</table>

Sapropel-lime-hemp composite SLHC 3-4	Sapropel Lime Hemp shives	Raw material ratio (filler:binder:binder)	1:5:5
		Density kg/m^3	533.58;540.59
		Thermal conductivity, W/m·K	0.089
		Mechanical strenght, MPa	0.77
Lime-hemp composite LHC 1-2	Lime Hemp shives	Raw material ratio (filler:binder:binder)	1:2.5:2.5
		Density kg/m^3	294.09;302.40
		Thermal conductivity, W/m·K	-
		Mechanical strenght, MPa	0.29
Lime-hemp composite LHC 3-4	Lime Hemp shives	Raw material ratio (filler:binder:binder)	1:5:5
		Density kg/m^3	498.32;562.93
		Thermal conductivity, W/m·K	0.099
		Mechanical strenght, MPa	0.90
Lime-hemp composite LHC	Lime Hemp shives	Raw material ratio (filler:binder:binder)	1:5:5
		Density kg/m^3	408.10
		Thermal conductivity, W/m·K	0.086
		Mechanical strenght, MPa	0.61
Sapropel-wood fiber composite SWF	Sapropel Wood fiber	Raw material ratio (filler:binder)	1:6
		Density kg/m^3	319
		Thermal conductivity, W/m·K	0.19
		Mechanical strenght, MPa	0.060
Sapropel-birch wood sanding composite SWD	Sapropel Wood sanding dust	Raw material ratio (filler:binder)	1:6
		Density kg/m^3	470
		Thermal conductivity, W/m·K	0.061
		Mechanical strenght, MPa	0.67
Magnesium-hemp composite MHC 1	Magnesium chloride Hemp shives	Raw material ratio (filler:binder:binder:binder)	1:1.25:0.9:1.33
		Density kg/m^3	302.3
		Thermal conductivity, W/m·K	0.076
		Mechanical strenght, MPa	0.25
Magnesium-hemp composite MHC 2	Magnesium chloride Hemp shives	Raw material ratio (filler:binder:binder:binder)	1:1.25:2:0.6
		Density kg/m^3	504.4
		Thermal conductivity, W/m·K	0.111
		Mechanical strenght, MPa	1.12

Obtained results show that the thermal conductivity of lime-hemp composite material (density 408.10 kg/m^3) is on average low - 0.086 W/m·K. Similar values are obtained for sapropel-lime-hemp material - 0.089 W/m·K. The results obtained are satisfactory, similar to those used in practice in the world for hemp shives, and with the current regulation, the wall insulated with such materials should be approximately 400 mm thick to reach regulatory values (Sinka et al., 2014).

In the study on sapropel and peat as a binder for wood chips (Obuka et al., 2014), the study also determined the mechanical strength of the materials. Depending on the amount of moisture, the compressive resistance of the board's changes. The compression resistance of sapropel-wood chips is 0.06 MPa and that of peat-swood chips is 0.13 MPa. In contrast, the bending resistance shows that the resistance of sapropel-wood chips is 0.02 MPa and that of peat-wood chips - 0.3 MPa. The results of compression and bending resistance show that the composite materials have sufficient strength to form adhesive joints and perform assembly work. Additional information about gained results is found - (Obuka et al., 2014).

The obtained results show that composite materials with filler of birch wood sanding dust and binder of green algae sapropel exhibit higher values in compression deformation perpendicular and parallel to the direction of specimen formation. The compressive results of perpendicular deformations range from 0.67 to 0.76 MPa. The result in linear deformations is 0.72 and 0.67 MPa, respectively. The results obtained from the compression resistance show that the materials are durable enough to be used in assembly work and to form adhesive joints. Additional information about test results is found in article - (Obuka et al., 2015).

3.3. Thermal conductivity test of composite materials

In a study on composite materials of sapropel as binder, a thermal conductivity test was performed by changing the types of sapropel and different filler materials (Obuka et al., 2015). Three types of sapropel from Lake Veveru (green algae) and Pilvelu (cyanobacterial algae) and a carbonate sapropel from Lake Padelis (carboniferous) were used. Fillers used were hemp fibers and shives, wood fiber, birch wood sanding dust. For the developed composite materials raw sapropel was used as a binder. The results obtained from the tests are shown in Table 3.3.

Based on the obtained results, it can be concluded that the material which contains wood fibers and sapropel of green algae from the Lake Veveru shows the best results. The results show that these composite materials have similar characteristics and thus similar applications and potentials. The composite materials produced have low thermal conductivity due to their mixed, fine-porous structure and have a homogeneous fiber structure with interconnected and open pores. Due to the organic origin of the raw materials, the composite material of the sapropel binder and hemp shives has a heterogeneous structure. The granulometric disintegration of different particles results in voids and uneven composition with weaker inclusions and faster deformation of the sample. Additional information about test results is found in article - (Obuka et al., 2015).

Table 3.3. Thermal conductivity results of composite materials

Material: binder-filler	Density, kg/m^3	Thermal conductivity, W/m·K
Carboniferous and green algae sapropel - hemp	191	0.063
Carboniferous and cyanobacterial sapropel - hemp	200	0.059
Wood fiber – green algae sapropel	153	0.055
Wood fiber – cyanobacterial sapropel	202	0.060
Cyanobacterial sapropel – wood sanding dust	214	0.061
Cyanobacterial sapropel – wood sanding dust – Aerosil silica	376	0.080

In the study on sapropel and peat as a binder for wood chips (Obuka et al., 2013), the thermal conductivity of the materials reached 0.067 and 0.060 W/m·K. The study takes into account the number of freezing cycles and the humidity of the materials tested. The results show that the thermal conductivity of peat and particleboard increases, while that of sapropel and particleboard decreases during the freezing cycles. Comparing the obtained results, where the moisture content of the composite material is from the air-dry state to the moisture-saturated material (12%), the thermal conductivity coefficient is 0.050-0.060 W/m·K for sapropel-wood chips and 0.055-0.064 W/m·K for peat-wood chips, respectively.

3.4.Compressive and flexural strength tests of composite materials

In this study, composite materials were developed using sapropel as an adhesive. Two types of sapropel samples were used - the green algae sapropel obtained at Lake Veveru and cyanobacterial sapropel obtained at Lake Pilvelu. Characteristics such as dry matter content, moisture content and density were determined for sapropel. Adhesives were tested by gluing plywood and mechanical testing of the material: bond strength test, static bending using (Anonymous, 2000b) and (Anonymous, 2005) standard, application group (D1 - D4) for sapropel as glue using (Anonymous, 2001a) and (Anonymous, 2002) standards, pulling peat with sapropel and determining strength perpendicular to the slab plane using (Anonymous, 2000a) standard.

The results show that the highest result in the mechanical bending test for the sapropel adhesive (Anonymous, 2001b) standard for the determination of elastic modulus and bending force was found in the Lake Pilvelu sapropel (88.7 MPa in parallel bending). The lowest result, however, is shown by the same type of sapropel only perpendicular to the bend (Fig. 3.2.). In addition to determining the applicability group for the sapropel as an adhesive (durability test), the Pilvelu sapropel - beech samples show a result of 3.67 MPa. In addition, tests according to the standard EN 319 were performed to detect dried natural peat and sapropel as a glue for tensile strength perpendicular to the grain direction, results indicated: samples of peat – Pilvelu sapropel - 0.077 MPa, samples of peat – Veveru sapropel - 0.067 MPa.

The results of the study indicate that an environmentally friendly adhesive derived from sapropel can be used as a natural binder in composite materials, which has a high adhesion and retention capability.

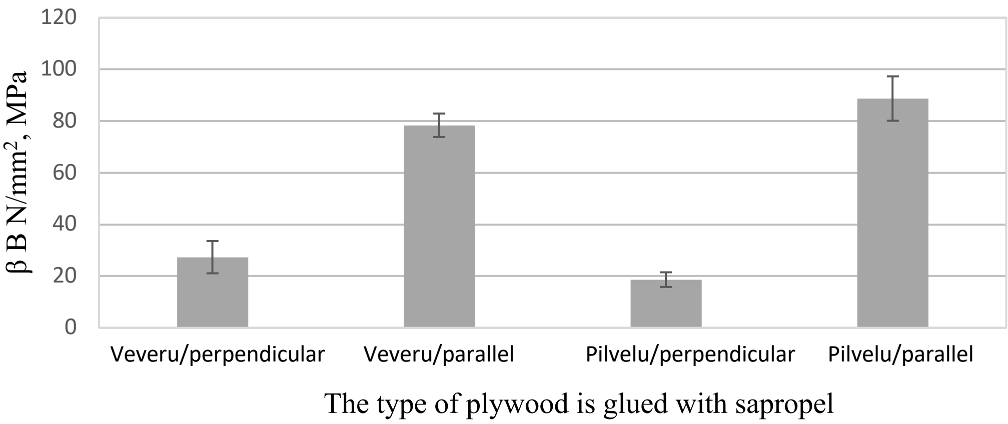

Figure 3.2. Bending mechanical strength in parallel and perpendicular to the fiber direction

The research direction is promising and should be continued to improve the properties of sapropel adhesive and to increase its effectiveness.

3.5. Sound insulation properties

Study on sapropel and peat as a binder for wood chips (Obuka et al., 2014) states that ability to insulate sound is one of the important properties of any building material. Thus a study of sound insulation properties of the developed materials was carried out within the framework of the thesis (Table 3.4.).

Table 3.4. Results of sound insulation tests using 4 microphone method

Tested plate	Sound insulation, dB
Peat - wood	30
Peat - wood	32
Sapropel - wood	32
Sapropel - wood	31

The obtained biocomposite materials have a fine-porous structure with a homogeneous fibrous structure with open and interconnected pores. The obtained sound insulation results indicate very good insulation properties of the composite material. Compared to other ecological thermal insulation materials, such as flax fiber thermal insulation material, the results are worse and differ by 14 dB. According to the literature, the sound insulation result of flax fiber material is 45 dB, but flax-wool thermal insulation material retains 40 dB sound absorption (Kozłowski et al., 2008). Given the sound insulation properties, it can be concluded that better sound insulation level can be reached using heavier materials.

3.6. Auto-ignition test

In the study on sapropel and peat as a binder for wood chips (Obuka et al., 2014). Auto-ignition risk assessment of the developed materials was performed. Based on the data obtained from the combustion test, it is possible to conclude that the auto-ignition temperature of peat particle board is higher than the auto-ignition temperature of sapropel-particle board. In order to improve the usability of sapropel based biocomposites, they must be treated with various means that improve fire safety and biological resistance, as this increases the durability and use of composite materials.

3.7. Evaluation of the biodegradability of the tested materials

Biodegradation experiments were performed by adding the composite materials to the soil amended with nutrients and a consortium of microorganisms with a high hydrolytic activity (Muter, 2015) in order to provide favorable conditions for degradation processes.

For the developed composite materials three types of raw sapropel have been used as a binder, i.e., green algae sapropel (GAS); cyanobacteria sapropel (CBS) and carbonatic sapropel (CS).

Fillers of biocomposite materials were used in the biodegradation test - wood fiber, birch sanding dust, hemp shives. Mineral binders developed in previous studies were used for these materials - dolomitic lime consisting of 100% DL60 lime (Dolomite) and hydraulic lime consisting of 60% DL60 lime and 40% calcinated kaolin clay (Clay) (Sinka and Sahmenko, 2015). Binder-filler mass ratio was 2:1. Block peat ("Laflora") was also used for composite materials biodegradation studies as a control material (Obuka et al., 2019).

3.7.1. Respiration intensity of microorganisms

The respiration intensity of microorganisms in the experimental batches was observed before and after 7-day incubation period at 37 °C. An increase of respiration intensity in the composite materials has been observed. The amended batches at the beginning of incubation showed statistically significant difference ($p<0.05$) and varied in the range from 31% to 70%, as compared to the control batch with soil and peat. For additional information about results in this study can be found - (Obuka et al., 2019).

The obtained data can be interpreted as the potential biodegradability of the tested composite materials under given test conditions. It shows that the materials used are biodegradable at a varying rate. It is seen that it is mostly dependent on the used filler. The wood sanding dust has the lowest biodegradability as it shows the lowest respiration after 7 days, while wood fibers and hemp shives have higher biodegradability as they have higher respiration after 7 days. The used materials demonstrate that with different extent all studied materials are biodegradable and can be used to decrease the overall environmental impact of construction materials.

3.7.2. Enzyme activity of microorganisms: fluorescein diacetate hydrolysis

One of criteria for evaluating the biodegradability of the tested materials could be an increase of microbial enzyme activity, which responded to the presence of bioavailable nutrients. FDA hydrolysis involves the activity of various enzyme groups of microorganisms, i.e., hydrolases, proteases, esterases, lipases, etc. (Green et al., 2006). For additional information about results in this study can be found - (Obuka et al., 2019).

Comparison of the FDA hydrolysis activity showed a statistically significant ($p<0.05$) difference between control and composite materials, except CS/wood fibers. Greater FDA hydrolysis activity may indirectly indicate more intense biodegradation processes, as it depends on the availability of nutrients, the concentration of microorganisms, and their physical, chemical and environmental properties (Green et al., 2006; Mupambwa and Mnkeni, 2016).

3.8. Sapropel, peat, biochar granules for the agricultural purposes

In this thesis part study, sapropel and peat are considered as potential soil improvers in the form of granules. Until now sapropel in Latvia was mainly used for fertilization of fields. In addition, sapropel can be used as a binder, for example in granules development for strength enhancement (Balčiūnas et al., 2016; Obuka et al., 2015; Vincevica-Gaile et al., 2019).

The aim was to find out the possibilities of peat-sapropel and sapropel granules formation and to evaluate their properties. During the development of the sapropel-peat and sapropel granules, the binder was machined prior to incorporation - compacted to a homogeneous mass. The study determined the physical and mechanical properties of test sapropel granules for three types of granules. Gravity density, water absorption test and granules compressive strength, environmental acidity response and electrical conductivity were determined.

Granules from pure sapropel and water pH = 7.35, granules from peaty sapropel and sapropel pH = 7.36, granules from sapropel-peat pH = 4.52 were made. The formed granules slowly decompose in the aquatic environment. In the soil environment, granule decomposition occurs as a result of physical action.

Gravity density of granules from pure sapropel and water is 639.6 kg/m^3, but granules from peaty sapropel - 246.1 kg/m^3, from sapropel - peat - 248.3 kg/m^3. The water absorption of granules from pure sapropel and water is ≤ 78.9%, granules from peaty sapropel - 167.8%, sapropel – peat granules - 163.9%. The mechanical strength of pure sapropel and water granules is 1.06 MPa, that of peaty sapropel - 0.46 MPa, that of sapropel-peat granules - 0.44 MPa. As a result, granules with sufficient mechanical strength for long-term storage, transport and incorporation into the soil were obtained.

Biochar-sapropel granules were investigated in thesis as well. The aim of the study was to investigate the possibilities of bio-char-sapropel granules formation and to evaluate their properties by using bio-char as a by-product in cogeneration plants. In the aquatic environment, granules decompose slowly because both ingredients are water insoluble. Therefore, their degradation in the soil environment is due to physical effects, but the degradation of both bio-carbon and sapropel is slow (De Gisi et al., 2014). The materials used are free of heavy metals and are considered safe for use in agriculture.

Bulk density of Lake Pikstere and biochar granules: by type of granules - cylindrical after extrusion (0.31 ± 0.07 g/cm^3), spherical after extrusion (0.47 ± 0.18 g/cm^3), spherical after rotation molding movements (1.00 ± 0.43 g/cm^3). The granules have a low bulk density compared to commercially available compressed biochar granules.

Analysed raw materials do not contain heavy metals and are therefore considered safe for use in agriculture.

3.9. Sapropel composite material microbiological stability study

Microbiological stability of sapropel and lime, magnesium oxide-chloride as binder for composite materials of hemp, sapropel as binder for wood fiber, wood sanding dust were studied.

This study used sapropel-lime, magnesium oxide-chloride, and lime binders; filler composite materials included hemp shives, wood fiber, and birch wood sanding dust. The fillers used are industrial and agricultural by-products that needs to be recycled or used repeatedly. Additive "ALINA LIFE^{TM} was used as an antimicrobial component. The samples were tested using the fungi *Alternaria alternata* and *Cladosporium herbarum.*

During the research it was found that the fungi of the genera *Sordaria*, *Alternaria* and *Fusarium* are the most common on the materials used. Isolated cases of *Penicillium*, *Acremonium*, *Paecilomyces*, *Trichoderma*, *Mucor* and *Stachybotrys* spp. - it indicates that the substrates contain sufficient moisture and nutrients for fungal development. Well-developed fungi were observed on sapropel-wood fiber and birch wood sanding dust materials, which can be explained by the fact that the pH level is neutral (pH = 6-7) or even slightly acidic (pH = 5), that wood is naturally a suitable substrate for many fungi.

Fungal development was practically non-existent on hemp-lime materials and hemp-magnesium chloride binder as well as hemp-sapropel-lime binder materials. This is due to the antimicrobial activity of cannabis (Ali et al., 2012), as well as the naturally high pH (pH = 9-12) of lime, which adversely affects fungal development.

The study proved that increased intensity of fungi growth occurs in materials that are made of wood fibre, wood dust and and sapropel as binder. It should be revealed that the fungi species that grow on the material depend on the type of material, surface area, filler and binder. Fungal growth was practically not observed on hemp-lime and hemp materials - magnesium chloride binder, as well as hemp-lime-sapropel materials. For additional information about results in this study can be found – (Obuka et al., 2017).

For the test part 2.1. of the experiment, the samples were prepared in 70×70×70 mm cube moulds, wood wool (WW) was cut with a similar surface area. For the second stage, the samples were prepared in 40 mm diameter and 10 mm high cylindrical forms, with wood fibre cement board (WF) and WW cut with similar surface area; for the test part 2.2., additional samples with lowered mineral binder amount were also produced, using 50% and 20% of the binder amount of the first stage with the same amount of shives, producing samples with less binder coverage and microbiological protection.

From analysing the changes in the microbiological stability of composites depending on the concentration of the binder (Fig. 3.7), it can be concluded that a decrease in the amount of mineral binder decreased microbiological stability. For MOC, FHL, and HL biocomposites at 100% concentration the fouling assessment was 0–1.5, at 50% concentration it was 1–3, and at 20% it was 2.5–4 (Fig. 3.7.).

The decrease in microbiological stability correlated with the lowering of pH in the specimens. Lime-based binder biocomposites (FHL and HL) showed higher microbiological stability than MOC biocomposites, since on the 100% binder specimens fouling was not observed, while on the MOC the fouling corresponded to the levels 1–2. At 50% and 20% specimens, this difference disappeared. This can be attributed to the pH level, which for the lime-based specimens was around 12 at 100% but for the MOC was 9.76, while the reduction of binder in 50% and 20% specimens resulted in a similar pH and microbiological stability.

The decrease in microbiological stability in the MPC biocomposites was less pronounced, as the intensity of growth at 100% concentration was 2–4, while at 20% it was 3–4. Such an increased intensity of growth in MPC was similar to the results of the first stage of the experiment and can be explained by the impact of the hardener, potassium phosphate.

In the second stage of the experiment, it was discovered that fungi belonging to the species of *Paecilomyces* and *Stachybotrys* were the most common on the materials included in the research. In some cases, *Penicillium, Acremonium, Cladosporium, Aspergillus, Trichoderma* and *Mucor* were also observed, indicating that the substrates contained sufficient amounts of moisture and nutrients for the fungal development. Most of these fungi feed on cellulose; therefore, they can be found on cellulose-based materials (Bech-Andersen, 2004; Klamer et al., 2004). *Stachybotrys* also feeds on lignin and for this reason, it is often found on wood and its products (Vance et al., 2016), and it is also known as black mould (Ding et al., 2018).

Microbiological stability tests have been developed for sapropel-based new materials for application of functional properties. Bio-based composite material, where sapropel was used as a binder, shows one of the highest microbiological stability results. In addition, fungi and other organisms were not detected on the samples.

The tested sapropel, lime and magnesium oxychloride cement bio-based composite material have a higher microbiological resistance than commercially used wood wool insulation; therefore, they have the potential to be used in construction under similar conditions, i.e. in structures protected from external moisture. Using visual expert conclusions, as in this study, can give a modest insight into the microbiological resistance and stability of the studied composite materials. It may also be that a fungus with a robust effect on the material can give a low growth percentage share. Organoclay-added samples showed 13.8% lower overgrowth and those with the biocide product had 9.1% lower, however incompatibility was observed with formulated hydraulic lime (20%), magnesium oxychloride cement (20%), hydraulic lime (20 %) and magnesium phosphate cement (100%) binders. For additional information about results in this study can be found - (Obuka et al., 2021).

Conclusions

1. Organic-rich sapropel is prospective material for diverse applications: it shows high prospects to act as biological glue combined with fibrous organic materials.

2. Using local resources such as sapropel and by-products of the production process, such as birch wood sanding dust, birch wood fibers, hemp shives and wood chips, it is possible to develop environmentally friendly composite materials in construction and agriculture, adapting them to the needs of use.

3. Biological stability of natural sapropel containing biocomposites is one of key parameters for their application potential and should include detailed evaluation of composites in respect to major groups of microorganisms of concern.

4. The mechanical and thermal properties of sapropel-based composites were similar to those of synthetic as well as mineral materials, suggesting that sapropel composites could have similar use in the construction industry: as a self-bearing wall thermal insulation material that works together with the structural timber frame.

5. As the sapropel-based building materials have high organic content, they are vulnerable to biodegradation; therefore, antimicrobial additives are significant to add.

6. Microbiological stability and biodegradation tests have been developed and adapted to apply the functional properties of sapropel-based biocomposite materials.

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