

Article



# Reutilization Prospects of Diamond Clay Tailings at the Lomonosov Mine, Northwestern Russia

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**Abstract:** Approaches to reutilization of diamond clay tailings in northern environments are considered in the example of the Subarctic region of Russia. The monitoring studies are conducted at storage facilities of Severalmaz PJSC where ca. 14 million cubic meters of waste rock are produced annually after kimberlite mining and processing. The tailings of diamond ore dressing waste are situated in complex geological conditions of high-groundwater influx and harsh cold climate with low levels of solar radiation and the average annual temperature below freezing point. Furthermore, the adjoining protected forests with a significant diversity of biogeocenoses and salmon-spawning rivers are affected by the storage area. Reducing the impact of the tailings can be achieved through the reuse of the stored clay magnesia rocks obtained from saponite-containing suspension. The experiments reveal the most promising ways of their application as potential secondary mineral raw materials: cement clinker and ceramics manufacture, integration of alkaline clay into the reclamation of acidic peat bogs, and production of aqueous clay-based drilling fluid. Field and laboratory tests expose the advantages and prospects of each suggested treatment technique.

**Keywords:** saponite; waste recycling; eco-friendly reuse; land reclamation; deacidification; Histosols; Technosols; drilling mud

# 1. Introduction

## 1.1. Mineral Processing Waste as An Environmental Issue

The extraction of low-grade and deep-lying mineral resources results in a gigantic increase in the volume of overburden and host rocks mined and stored on the Earth's surface. Waste accumulation leads to dangerous environmental contamination, wasteful use of natural resources, significant economic damage, and poses a real threat to the health of present and future human generations. Thus, the issue of mining and processing left-overs and their impact on the adjoining landscapes is one of the most acute for the commodity sector, and the development of novel waste management technologies is particularly urgent.

In the areas of mining and processing enterprises, storage facilities of mineral waste are among the main sources of disturbance and pollution of all the media of the natural environment [1–3]. Up to the present time, the dominant waste management technique of the mineral resource complex is surface storage, affecting the ecological situation over areas of millions of hectares, transforming ecosystems, worsening the sanitary and hygienic situation, and destroying natural resources [4,5].

## 1.2. Mineral Processing Waste as A Promising Raw Material

The management of the solid part of tailings storage facilities is among the most important environmental challenges of mining operations. Oftentimes, these residuals pose a great threat because

of the geochemical properties; the immediate measures are to be taken in such cases [1]. Worryingly, when macro- and micro-components are found in non-hazardous concentrations, fewer efforts are put into the environmental management of the tailings [6], though technogenic sediments offer prospects for reuse and valorization beyond their traditional disposal.

Argillaceous minerals are reckoned among these materials, and saponite is a demonstrative example of the tailings constituent that is often left unfairly mistreated [7]. According to the International Mineralogical Association list, its formula is  $Ca_{0.25}(Mg,Fe)_3((Si,Al)_4O_{10})(OH)_2 \cdot nH_2O$  [8]. Although being regularly safe in terms of chemical contamination, saponite tailings may pollute the air as a result of fugitive dust emission, damaging flora and fauna and posing a danger to human health [9]. In water ecosystems, saponite forms intermolecular associates with dissolved organics and then settles out, creating favorable conditions for the introduction of invasive species [10]. Studies show that saponite reutilization is not only a question of eco-risk abatement but also an economically reasonable decision as the mineral is a valuable secondary product with a wide range of applications, which include the chemical, food and consumer goods industries, agriculture, medicine and pharmacology, foundry practices, metallurgy, and construction [11].

The high sorption capacity of saponite brings an opportunity of its use as a geochemical barrier. The mineral has been recently applied for the removal of the  $Cu^{2+}$  ions from aqueous solutions by using its natural and acid-activated forms [12], as well as by mechanically (via high-energy grinding in a planetary ball mill) activated samples for the Cu<sup>2+</sup> and Ni<sup>2+</sup> ions adsorption [13]. The original technique allows Cr<sup>6+</sup> removal from water by natural ferro-saponite [14]. A composite containing saponite, an organic ion, and a metal-binding agent has eliminated metal pollution in several experiments [15]. However, the review has shown that further research is needed on the modification and synthesis of new saponite materials for adsorbing of dissimilar heavy-metal pollutants from the environment [16]. The saponite-based sorption technique can be likewise applied in the purification of uranium-contaminated water; the process has been affected by such complexing reagents as EDTA, carbonate ions, and fulvic acids [17]. Quantitative determination and comparison of the reactive properties of saponite and bentonite in europium immobilization have revealed that the reaction rate is independent of the clay type, but the immobilizing ability of bentonite is lower than that of saponite [18]. Niobium saponite clay has even been suggested for the catalytic oxidative abatement of chemical warfare agents [19]. Carbon capture using amine-modified saponite has been proposed to reduce the  $CO_2$ atmospheric concentration after hydration and functionalization by grafting with aminopropyl and diethylenetriamine organosilanes or impregnation with polyethyleneimine or double functionalization by impregnating previously grafted samples [20].

The pathway towards the modification and hybridization of saponite for composite and nanocomposite production has shown itself in the best light [21]. The multifunctional synthesized nanocomposites can include the layers of anionic clay intercalated between the layers of cationic clay [22]. The proposed modified polymeric saponite [23] and poly(vinyl chloride) nanocomposites [24] show thought-provoking thermal behavior. The advantages of microwave synthesis yielding mesoporous acid saponite characterized by a surface area of 603 m<sup>2</sup>/g and lamellae crystallite size of about 4 nm have been demonstrated by using a quaternary ammonium salt and surfactants or polymer [25]. Furthermore, the Cu-saponite catalysts have been proposed for soot combustion [26].

Modified and virgin saponite is being increasingly used in the construction material manufacturing. Its suitability as the main binder for the alkali-activation process for the fireplace element production has been positively tested using sodium silicate and hydroxide solutions as activators [27]; another study has confirmed saponite usefulness in thermal insulation composites [28]. In much the same way, it has been recommended as a binder in the manufacturing of steam-cured silicate materials [29]. Highly-dispersed saponite-containing material is suitable for concrete modification [30] and the water-cement ratio in mortar has been successfully controlled; the concrete strength has been raised 2-fold and the frost-resistance quality has been improved by adding 7% of saponite [31]. Likewise, the plastering mortar from natural minerals, including saponite, has been characterized by low fissuring

and better acoustic absorption, adhesion, and initial strengths [32]. Modified saponite-containing product has been effectively used to produce high-quality ceramic bricks; the sample density amplified with the increase of sintering temperature:  $1.9 \pm 0.1$  g/cm<sup>3</sup> (800 °C)– $2.2 \pm 0.2$  g/cm<sup>3</sup> (1000 °C) [33]. Furthermore, the mineral has been reutilized from waste to pelletize iron-ore concentrates and produce high-quality building materials [34].

The related advantageous field of saponite reprocessing is the aqueous solution preparation. For instance, liquid suspensions of water, alcohol or carbonic acid have been stabilized due to the presence of fine (3 mm to 50  $\mu$ m) saponite, which makes it possible to avoid sludging during the pipeline or cistern transportation [35]. A recently developed aqueous composition for surface friction reduction contains a solid lubricant and a modified, or synthetic, clay mineral such as saponite, montmorillonite, and hectorite [36].

The mineral is medicine-applicable in nanocomposites consisting of quaternary fulvic acid and saponite, possessing high bacteriostatic activity [37]. Ferroan saponite with admixtures of quartz, feldspar, and calcite as well as exposed or hidden (layered at inner regions) nano-iron has proved its antimicrobial properties against Gram-negative antibiotic-resistant bacteria [38]. Saponite has even been used for the treatment of chronic prostatitis and erectile dysfunction [39].

We examined tailings storage facilities in Northwestern Russia at one of the world's biggest diamond mines [40]. A performed case study of kimberlite processing waste was purposed at the assessment of the reutilization prospects of saponite in a sparsely-populated harsh-climate area. The regional conditions complicate any reutilization attempts significantly and require a special approach, which takes into account the landscape-forming factors (low levels of solar radiation and the average annual temperature below the freezing point) and remoteness from consumer markets.

#### 2. Materials and Methods

## 2.1. Mining and Processing Plant

Europe's largest primary diamond deposit, Lomonosov, in the Primorsky District of Arkhangelsk Oblast (Figure 1a) is an area of intensive accumulation and storage of processing waste at the tailings (Figure 1b) of the Severalmaz enrichment plant. Being mined since 2005, the Arkhangelskaya pipe reached industrial volumes of 4 million tons of ore per year by 2013. Between 2014 and 2016, mining of the additional kimberlite pipe (Karpinskogo-1) and the completion of the new processing plant increased annual diamond production at Lomonosov from approximately 500,000 to 2 million carats [41,42]. Production is expected to increase to 5 million carats after the Pionerskaya and Lomonosovskaya pipes are developed [43].



**Figure 1.** Study area: (**a**) the Lomonosov deposit in Arkhangelsk Oblast (based on the free blank map, commons.wikimedia.org); (**b**) waste rock dumps and tailings storage facilities eastwards of the open-pit mines at the Arkhangelskaya and Karpinskogo-1 pipes (based on the Bing satellite image).

Mining operations are carried out classically, without the use of drilling and blasting, by means of hydraulic excavators at the Arkhangelskaya and Karpinskogo-1 pipes. Overburden and ore mined from quarries are transported by dump trucks to waste dumps and ore stockpiles where the kimberlite from both pipes is mixed to obtain uniform grades. After the weathering in the stockpiles, the ore enters the grate of the receiving hopper at the processing plant; prills of the average 0.4–0.7 m diameter are transported to wet autogenous mills. The initial processing phase tumbles the kimberlite in a large rotating mill that breaks the material into smaller pieces measuring about  $120 \times 25$  mm while mixing this material with water [44]. After milling, the material is dispatched to hydraulic separators that segregate likely ore material from waste material based on density. The crushed ore passes through the discharging grating of the mill to the screw-type classifiers where sludge is separated from the diamond-containing granular material. Utilizing hydraulic transportation, sludge reaches the tailings storage facility in the Zolotitsa river basin at the distance of 1.5 km from the processing plant. The remaining milled rock mass is sieved at high-frequency screens into three size classes and diamonds are identified by X-ray fluorescence and X-ray luminescence units [44]. Ore dressing produces 3.5 million m<sup>3</sup> of sludge and requires a large volume of water. Therefore, the enterprise provides its reverse supply to the concentration plant: suspended solids are gradually settled at the tailings, and water is then again involved in the enrichment process.

The forecasted mass of saponite to be discharged into the tailings is 68 million tons if mining to the depths of 460 m. Reserves for two pipes have been determined down to 460 m, yet Severalmaz's current plan is to complete open-pit mining on them by about 2026 when Arkhangelskaya is down to 324 m (currently at 154 m) and Karpinskogo-1 down to 260 m (currently at 105 m); a decision will then be made about whether it is more profitable to continue mining deeper or to mine one of the other pipes such as Pionerskaya [44]. One way or another, millions of tons of enrichment waste are to be produced in the upcoming years.

The Severalmaz tailings are, on the one hand, an environmentally hazardous object, and on the other, a technogenic diamond deposit, since kimberlite enrichment technology assumes that diamonds under 1 mm are not identified and are sent directly to the tailings [44]. Provided that the quarry develops the reserves to a depth of 460 m, ca. 9000 thousand carats of diamonds smaller in size than the criterion level—which are not taken into account by the state balance—are to be buried [7]. Although impressing, these numbers are less terrifying when compared with the total reserve estimated from all six pipes in the Lomonosov deposit that is approximately 167 million carats [44].

## 2.2. Study Area

The study area of Severalmaz is characterized by complex geological conditions [45,46] and high-water influx. The gentle undulating terrain is the prevailing land topography form, with a general southeastern slope; the absolute surface elevation is 90–150 m. The regional climate is fairly severe, with the lack of solar radiation and cold, long winters and cool summers; the average annual temperature is -0.6 °C, varying from -3.2 °C to +1.9 °C. Histosols, acidic peat soils, are formed under waterlogged conditions of the region that are typical of peat bogs, moors, and swamps throughout the taiga forest. The mining and processing site adjoins the protected forests with a significant variety of biogeocenoses and the salmon-spawning Zolotitsa river. Eco-activists claim that the pollution caused by diamond mining and beneficiation threatens to strip the upper Zolotitsa region of its environmental assets. Despite the low population density of 2.0/km<sup>2</sup>, territorial residents are actively using the natural resources in the traditional local industry [47]. This can raise an issue of compensation for indigenous people, such as in the case of the similar industry in the Sakha Republic [48,49].

The research findings confirmed that kimberlite processing implicitly results in the landscape's disturbance (the area of the tailings storage facilities is over 5 km<sup>2</sup>), pollution of the atmosphere, water, and soil, as well as the threat to the flora and fauna of the province. Saponite is the prevailing mineral of the tailings [7,9]. It interacts with components of bog ecosystems, changing their physicochemical properties and forming intermolecular associates with dissolved organic matter; this contributes to the

transport of mineral particles over long distances with the following sedimentation and the prolonged desorption, which creates favorable conditions for the introduction of invasive species [10]. With the fugitive emission from the open beaches and sides of the tailings dam, ca. 100 tons of inorganic dust (20–70% SiO<sub>2</sub> content) get into the air annually, which leads to the formation of an atmochemical pollution plume over an area of 15 km<sup>2</sup>. A significant proportion of the dust is deposited on high-value forest landscapes, causing substantial transformations [50–52]. Furthermore, the Global Tailings Portal, launched in January 2020 with support from the UN Environment Program [6], put the facilities into the Class 3 of 5—potentially dangerous objects, i.e., any accident might cause a disaster of more than local scale, according to the Russian EMERCOM classification [53]. Reducing the technogenic load of the enterprise on the environment and emergency prevention can be achieved through reducing the tailings area by developing novel waste reutilization approaches, largely dependent on the waste composition.

## 2.3. Slurry Sampling and Analysis

The sampling procedure was planned and completed according to the Russian national standard GOST 32026-2012 'Clay raw materials for manufacturing of clayite gravel, rubble and sand. Specifications'. To determine the reusability of enrichment waste, solids of the tailings were sampled at different depths (3.0; 3.5; 4.0; 4.5; 5.0; 5.5; 6.0; 6.5 and 7.0 m) in 10 uniformly distributed transects within the tailings' ponds. The mass of a spot sample was not less than 7 kg. Spot samples of each transect were thoroughly mixed and then averaged by the quartering method to obtain a combined sample. The mass of a combined sample intended for testing was at least four times the mass of a spot sample. After compiling an averaged specimen series of technogenic sediments, an analytical sample was taken from the combined sample by the quartering method for each analysis or experiment. The comprehensive laboratory studies determined the chemical and physical properties of the slurry samples at the Common Use Centre of the Saint Petersburg Mining University.

The crystalline phases of the tailing samples were identified using X-ray diffraction (*XRD*-7000, Shimadzu, copper target, continuous, scanning range 5–80°, scan rate 3.0°/min). The phases identification was performed using the International Modified Reference Powder Diffraction File (PDF) and the database of the American Society for Testing and Materials (ASTM). The content of rock-forming elements was determined by X-ray fluorescence spectroscopy on an XRF-1800 (Shimadzu) spectrometer for 4 averaged samples using a method with a double experiment repetition. To control the accuracy of the analysis, standard samples of the composition of clay and dunite were used. To determine the content of heavy metals in solid mineral waste, the HCI-extracts were analyzed by the atomic emission method using an induction-coupled plasma spectrometer (ICPE-9000, Shimadzu). The particle size distribution analysis of the tailings was carried out using a laser diffraction particle size analyzer (LA-950, Horiba).

After obtaining the data on the key slurry properties and thoroughly considering the most promising fields of saponite slurry reutilization, we have formulated a hypothesis that three application methods are feasible in the Subarctic region of Russia and are relevant for the needs of Arkhangelsk Oblast:

- Cement clinker and ceramics manufacture
- Integration of alkaline clay into the reclamation of acidic peat bogs
- Production of aqueous clay-based drilling fluid

A set of experiments was conducted to test the hypothesis and evaluate the potential technological capability of the implementation of the proposed procedures on waste reutilization prospects.

#### 2.4. Thermal Treatment and Compression Tests

Puck-shaped bricks with a diameter of 32 mm and a height of 32 mm were made from a total clay mass by a compression ring. Samples were backed in a muffle furnace on a phased basis: (i) drying at 120 °C for initial water vaporization, 10 h; (ii) dehydration at 600 °C to remove chemically bound

moisture, 5 h; (iii) firing at 800, 900, and 1000 °C, 4 h; and (iv) cooldown to a temperature of 50 °C, 5 h. After the thermal treatment at different temperatures, 5 sample series were obtained. The resulting bricks were subjected to the uniaxial compression using the Testometric testing machine under the maximum pressing force of 500 kH. The experiments have been done in triplicates to ensure the quality of the results.

## 2.5. Soil Deacidification Experiments

In the framework of experiments to study the properties of clay slurry, peat extract was treated with the saponite-containing pulp. The exchangeable acidity of peat refers to the number of absorbed acid cations, aluminum, and hydrogen, which can be determined by displacing into the liquid phase by neutral salts (primarily potassium chloride KCl). Ions of hydrogen and aluminum, which passed from a bound state to a dissolved one, are harmful to cultivated plant species; for that reason, this parameter is strictly controlled. Analysis of pH<sub>KCl</sub> allows soil classification by this criterion.

The peat extract was made based on moist saturated peat (humidity of 70%) weighing 5 g with the 1:2.5 ratio of dry substance to solvent. A 100 mL aliquot of 1 mol/dm<sup>3</sup> KCL solution was added to the unfiltered extract according to the procedure of the Russian national standard GOST 26483-85 'Soils; Preparation of salt extract and determination of its pH'. Measurement of pH was carried out using an Expert-001 magnetic stirring pH ionomer.

As a common initiative of the Department of Geoecology of the Saint Petersburg Mining University and the Department of Geology and Mining Operations of the Northern (Arctic) Federal University, an experimental site was established in the adjacent territory to study the ecological properties of tailings. Conditions of the practically in situ experiment were close to natural for modeling the natural settings of peat neutralization by saponite in open-air wooden boxes. The boxes were assembled and planted triplicate. Deacidification was carried out under 9 different ratios of natural to technogenic materials in 5 series of experiments: 90% peat extract/10% pulp, 80%/20%, 70%/30%, 60%/40%, 50%/50%, 40%/60%, 30%/70%, 20%/80%, and 10%/90%.

## 2.6. Drilling Fluid Quality Evaluation

To determine the suitability of the clay suspension of the pond zone for use as a drilling fluid, it is necessary to determine the density, viscosity, filtration rate, and gel strength. The experiments have been done in triplicates. Determination of the assumed viscosity was conducted using an SPV-5 fluidity meter by the outflow time of 500 cm<sup>2</sup> of the clay slurry fluid through a vertical tube in thrice-repeated series of measurements. The gel strength of drilling fluid, i.e., a measure of the inter-particle forces that indicates the gelling that will occur when circulation is stopped and prevents the cuttings from setting in the hole, was measured at a low shear rate after the drilling fluid was static for 10 s and 10 min using an SNS-2 device. Water loss was determined on a VM-6 device that measures the friction coefficient of the crust formed after filtering the drilling fluid by three main components: a bracket, a filter cup with accessories, and a pressure cylinder. The water loss was measured at a temperature of 20 °C by the volume of filtered liquid from the solution (cm<sup>3</sup>) in 30 min at an overpressure of 0.1 MPa with a filter area of 44 cm<sup>2</sup>, 75 mm in diameter.

## 3. Results and Discussion

## 3.1. Composition of the Diamond Clay Tailings

The Severalmaz tailing storage facility consists of the beach area and the core pond part (Figure 2a). The laboratory studies revealed the nature of the solid component of the technogenic material (Figure 2b) and the composition of the particle size fractions. The beach area is composed of sandy reddish-brown unconsolidated sediments. Quartz (up to 80%), dolomite (about 10%), and calcite dominate in the mineral composition. The saponite content in the sand of the beach zone reaches 3%. Particles of the beach area are changing from fine sands in the prismatic part of the beach area, to silts in the marginal

parts of the beach area near the water's edge. Bottom sediments in the pond sector of the tailings are represented by reddish-brown clay consisting of saponite (up to 70%), quartz (10–12%), calcite (8–10%), dolomite (2–4%), and illite (3%). The main pond part of the tailing dump is filled with the liquid component of the pulp. In the upper part of the pond zone (to a depth of ~1.5–2.0 m), water practically does not contain solids; an underlying stratum of an aqueous suspension (gel) is made up by saponite (up to 90%), quartz (5%), illite (3%), and kaolinite (2%). The discovered fluctuations in the concentrations of saponite in the bottom sediments of the pond part in the range up to 70% can be explained by the uneven composition of the kimberlite extracted at the Arkhangelskaya and Karpinskogo-1 pipes; saponite content in suspension is up to 90%.



(a)

(b)

**Figure 2.** Kimberlite processing waste: (a) the settling pond and solids of the beachside terrain; (b) close-up view of the characteristic slurry with the predominant (85%) particle size class of  $<25 \mu m$  [54].

The environmental safety and compliance of the wastes are the primary parameters determining the slurry reutilization prospects, as mining and processing impact is fraught with the land pollution issues that are to be abated [55,56]. In this context, we studied the content of potentially toxic elements in five averaged samples of the aqueous suspension (gel) from the pond zone. The ratio of heavy metal content in the solids of the tail suspension to the maximum permissible concentrations in soils (Table 1) showed that the levels of elements do not exceed the thresholds [57]. Thus, the products obtained from the Lomonosov diamond ore dressing waste are harmless to humans and comply with sanitary standards.

| Sample | Cd   | Со   | Cr   | Cu   | Ni   | Pb   | Sr   | Zn   |
|--------|------|------|------|------|------|------|------|------|
| SP-1   | 0.25 | 0.26 | 0.21 | 0.90 | 0.47 | 0.01 | 0.26 | 0.78 |
| SP-2   | 0.20 | 0.28 | 0.26 | 0.76 | 0.50 | 0.03 | 0.52 | 0.95 |
| SP-3   | 0.35 | 0.30 | 0.24 | 0.86 | 0.51 | 0.02 | 0.25 | 0.86 |
| SP-4   | 0.22 | 1.20 | 0.23 | 0.83 | 0.49 | 0.01 | 0.27 | 0.82 |
| SP-5   | 0.25 | 0.28 | 0.21 | 0.80 | 0.52 | 0.03 | 0.26 | 0.95 |

**Table 1.** Ratio of the measured metal concentrations in the saponite pulp samples (SP) to the maximum permissible concentrations in soils.

Another issue that can restrict waste reuse is the technogenic diamond deposit in the tailings. The analyzed characteristics of the mineral phases of the enrichment waste from the crater part of the Arkhangelskaya pipe allow the conclusion that the greater part (over 90%) of the substandard diamonds discharged into the tailing dump will be concentrated in the sediments of the beach area, a minor part will be in bottom sediments, and a negligible amount in the saponite suspensions [7].

Thus, saponite meets the environmental requirements as secondary raw material, and its selective extraction from the tailings pond can significantly reduce the environmental burden, primarily due to the reduction of the tailings pond area itself and the elimination of the risk of breaking through the dam walls. The reprocessing of low-grade (in terms of diamond content) saponite suspension for diamond recovery would not be economically feasible. On the contrary, other application scenarios can be considered as "beneficiation" of the remaining technogenic diamond deposit in the sediments of the beach area and the bottom sediments.

#### 3.2. Reprocessing as Building Materials

Since enrichment waste consists largely of saponite, which is a clay magnesian raw material, it can be disposed of in the production of building materials such as ceramic bricks, ceramic and pressed wall materials, and cement clinker [58–60]. The production of ceramic bricks is promising for the region, as the nearest large-scale production is in the city of Yaroslavl, ca. 1000 km away, from where the brick is imported into the Arkhangelsk Oblast. Ceramic and pressed wall panels can be produced from various types of mineral raw materials, e.g., gypsum or clays. To reutilize the waste from the Lomonosov deposit, Portland cement is to be added as a binding component of soft and fluid clays. As cement is always in demand in the construction industry, its production is relevant, both for the needs of the enterprise itself and for the sale of products [61]. The manufactured cement clinker can be used to produce cement at the Wall and Construction Materials Integrated Plant in Arkhangelsk, ca. 100 km away.

For the firing of ceramic bricks, the samples are obtained by the method of semi-dry molding, which is currently one of the most progressive approaches. The experiments on baking and testing of the samples for uniaxial compression showed that the temperature of 900 °C is required to increase the strength of a brick (Table 2). However, it is acceptable to carry out firing at a temperature of 800 °C with a slight decrease in strength. A further increase in the firing temperature leads to an increase in the fragility of the samples and strength reduction (Figure 3).

| Sample<br>Series                            | Brick firing<br>Temperature, °C              |                     | С     | ollapse Pre  | ssure, MI    | Pa                  |            | Average,<br>MPa |
|---|--|---------------------|-------|--|--------------|---------------------|------------|-----------------|
| SB-1  | 120  | 2.00                | 2.36  | 2.11   | 2.24         | 2.27                | 2.19       | 2.63            |
| SB-2  | 800  | 11.81               | 12.17 | 8.70   | 8.08         | 14.28               | 12.60      | 13.53           |
| SB-3  | 900  | 12.44               | 13.18 | 16.02  | 9.95         | 9.95                | 13.01      | 14.91           |
| SB-4  | 1000   | 8.08                | 9.70  | 8.83   | 11.44        | 10.07               | 9.62       | 11.54           |
| 16<br>Collapse pressure, MPa<br>4<br>0<br>0 | 0<br>200 400 600 8<br>Brick firing temperatu | 00 1,000<br>Ire, °C | 1,200 | 12<br>Collabse bressmre ; MDa<br>8<br>4<br>0<br>0<br>0 | 5 10<br>Soli | 15<br>ds content, 9 | 20 25<br>% | 30              |
|   | (a)  |                     |       |  |              | (b)                 |            |                 |

**Table 2.** Measured collapse pressure values by subjecting four saponite brick sample series (SB) to the controlled compressive displacement along a single axis.

**Figure 3.** Experimental curves of the brick strength properties as a function of (**a**) baking temperature; (**b**) total weathered solids, viz. saponite, quartz, montmorillonite, dolomite, calcite, hematite, K-feldspar, and almost certainly a talc-pyrophyllite mineral [54].

For the brick manufacture, the clay suspension of the pond zone and a minor part of the tailings beach can be reutilized. To mathematically substantiate the dependence of the strength of the samples on the firing temperature, a regression calculation is performed to obtain a correlation coefficient. A level of -0.74 confirms a high degree of inverse dependence of the strength of ceramic samples on the solids content, and, accordingly, direct dependence of the strength on the silt fraction content. The obtained samples confirm the hypothesis on the prospects of brick manufacture [62,63].

In further experiments, cement clinker was obtained; to obtain Portland cement, we propose preparing the raw material by finely grinding and mixing the components, 70–75% of limestone, 30–25% of clay, as well as possible additives. The raw mix can be fired using the capacities of the mentioned plant in Arkhangelsk loaded by no more than 20%, or after building in rotary kilns at the mine site. The prepared pulp is to be dehydrated before entering the rotary kiln. This reduces fuel consumption by 20–30% compared to the "wet" method; however, due to the work of dehydration plants, energy consumption increases, i.e., the energy intensity of production in total, remain on equal levels [64]. Wet preparation is optimal for soft raw materials of significant humidity: fine grinding and mixing of limestone and clay are carried out in the water, so the raw material mixture is in the form of liquid sludge with a water content of up to 35–45%. In our case, it is possible to immediately mix the pulp with limestone, which greatly simplifies the process. To recycle diamond industry waste by producing cement clinker, thickened tailings are to contain no sulfur and phosphorus impurities; if the condition is met, burning is carried out in the presence of water at a temperature of 1400–1500 °C with the addition of limestone (70 wt%) and bottom ash (5–10 wt%) to the slurry (20–25 wt%). Bottom ash additive, obtained from the slag fields of thermal power plants, reduces the moisture content of cement slurry and fuel consumption for clinker burning, thereby increasing the productivity of furnaces [65–67].

#### 3.3. Integration into Reclamation Scenarios

We used the enrichment waste as a fertilizer by analogy with dolomite and lime flour, i.e., with the use of crushed solid tailings sediments. Tail grinding occurs during the preparation of the raw material to obtain Portland cement, so there is no need for additional crushing operations, and, during one production process, two different technical problems are solved [68].

Peat soils (Histosols) are formed by the natural accumulation of partially decayed biomass and are typically acidic. To assess the regional properties of the predominant soil, we conducted a tenfold pH measurement in the peat extract (Table 3).

| Sample            | HE-1 | HE-2 | HE-3 | HE-4 | HE-5 | HE-6 | HE-7 | HE-8 | HE-9 | HE-10 |
|-------------------|------|------|------|------|------|------|------|------|------|-------|
| pH <sub>KCl</sub> | 3.54 | 3.55 | 3.73 | 3.68 | 3.69 | 3.54 | 3.54 | 3.50 | 3.49 | 3.55  |

Table 3. Ultra-acidic and extremely acidic pH of the Histosol KCl-extract (HE).

The pH range of the natural Histosols is ultra-acidic to extremely acidic [69,70]. Heather vegetation predominates in the turf layer of the site. Sedges, sphagnum, and woody residues are the peat-forming materials, in the top 25–30 cm horizon. The decomposition degree, i.e., the content of an unstructured mass that has lost its cellular structure, is 10–15%; according to the classification, peat belongs to poorly decomposed [71]. The total primary and secondary ash content is 4.3% and the density is 0.960–1.010 g/cm<sup>3</sup>.

Following the same procedure, we determined the pH of the saponite slurry (Table 4), and the strongly alkaline media was found, which corresponded to the statistics on the acidity of saponite materials [72,73].

 Sample
 SP-1
 SP-2
 SP-3
 SP-4
 SP-5

 pH
 8.72
 8.84
 8.95
 8.47
 8.80

Table 4. Moderately alkaline and strongly alkaline pH of the saponite pulp (SP).

Over the course of 5 experiments (Table 5), neutralization was carried out in 9 different ratios of the peat extract to the pulp. The obtained pH of the created Technosol varied between moderately acidic and moderately alkaline [74]. As soils developed under northern taiga vegetation, on the saponite predominating in the eluvium of mafic rocks, showed rather high landscape sustainability [75], we believe that these levels are satisfactory.

**Table 5.** Mass ratio of the Histosol KCl-extract (HE) to the saponite pulp (SP) and pH of the created series of Technosol mixtures (TM).

| HE/S | P Ratio | 90/10 | 80/20 | 70/30 | 60/40 | 50/50 | 40/60 | 30/70 | 20/80 | 10/90 |
|------|---------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
|      | TM-1    | 5.80  | 6.50  | 7.04  | 7.40  | 7.50  | 7.58  | 7.70  | 7.68  | 7.93  |
|      | TM-2    | 5.96  | 6.47  | 7.14  | 7.46  | 7.53  | 7.60  | 7.64  | 7.71  | 7.90  |
| pН   | TM-3    | 5.87  | 6.48  | 7.09  | 7.43  | 7.51  | 7.59  | 7.68  | 7.69  | 7.91  |
|      | TM-4    | 5.96  | 6.50  | 7.13  | 7.47  | 7.53  | 7.61  | 7.65  | 7.72  | 7.90  |
|      | TM-5    | 5.89  | 6.46  | 7.08  | 7.43  | 7.49  | 7.62  | 7.67  | 7.70  | 7.92  |

Figure 4a shows the smooth deacidification curve that confirms the regularity of acid peat reclamation with the alkaline pulp. The mixture reaches a neutral reaction even when 30% of the pulp is applied, which is a positive point since the natural material remains the major part constituent of the soil [76,77].



**Figure 4.** Experimental reclamation of Histosols: (**a**) deacidification curve of pH plotted against the mass ratio of the peat soil KCl-extract to the saponite pulp; (**b**) fertility levels of the created Technosols as a function of the tailings material mass fraction—practically bare surface, projective cover <0.25 at the sites TP-1, TP-2, TP-10, and TP-12; sporadic vegetation, projective cover 0.25–0.50 at the sites TP-9 and TP-11; sparse vegetation, projective cover 0.50–0.75 at the sites TP-7 and TP-8; and abundant vegetation, projective cover >0.75 at the sites TP-3, TP-4, TP-5, and TP-6.

To study the ecological properties of tailings, an experimental area was established in the adjacent territory, on the mesotrophic transitional-type swamp. We created the fertile Technosol by introducing the dehydrated clay pulp and peat mixture and cultivating plants suitable for reclamation of disturbed lands, as well as crops to substantiate the properties of saponite as an amendment and

fertilizer interchangeably with dolomite and lime flour [78]. The experimental site was arranged for several years.

Figure 4b depicts a schematic representation of the experimental squares into which the site was divided, indicating the percentage of dehydrated suspension in the soil and fertility levels of the material. The boxes were assembled and planted triplicate (Figure 5). Different blends of peat and clay pulp of the tailing dump were seeded with a proportional mixture of meadow bluegrass and fescue.



(a)



(c)



**Figure 5.** Experimental plots for perennial grass cultivation as a part of the reclamation procedure in the transitional peat bog within the boundaries of the Severalmaz allotment: (**a**) a fragment of the test site prior to herb sowing; (**b**) poorly decomposed top 25–30 cm Histosol horizon; (**c**) a separate bed of a control sample mixture of peat and enrichment waste; (**d**) a fragment of the experimental plot with perennial grasses in one year; (**e**,**f**) separate beds with different grass responses to growing conditions.

The results of the soil amendment experiment show the effectiveness of this saponite reutilization approach that gives the way to industrial implementation. We discussed the case within the framework of the Working Group on Land Reclamation, Environmental Protection, and Best Available Techniques (BAT) in Mining, a part of the Russian-German Raw Materials Forum [79], and recommended reusing saponite in this eco-friendly direction. Scaling up this technique from the local to a regional extent can contribute to the company's public image. Although land amelioration does not replace the need for compensation for indigenous people, the relationship with the local community can benefit from such practices [80].

## 3.4. Production of Aqueous Clay-Based Drilling Fluid

Drilling fluid, also called drilling mud, is a heavy, viscous mixture used to carry rock cuttings to the surface, to lubricate and cool the boring unit, and additionally to prevent the collapse of unstable strata into the borehole and the influx from water-bearing strata. A water-based drilling fluid typically contains clay to give it enough viscosity to carry cutting chips to the surface; other important properties dependent on this additive are density, filtration rate, and gel strength.

Viscosity is an indicator determining not only the magnitude of hydraulic resistance in the circulation system of a well but also penetration depths of flushing fluid into the pores and cracks of

rocks. With increasing viscosity, the conditions for cleaning the well from sludge deteriorate, and the mechanical drilling speed drops sharply. The average outflow time of 500 cm<sup>2</sup> of the clay slurry fluid through a vertical tube in thrice-repeated series of measurements was 1 min 11 sec. The clay slurry of the pond zone had the optimal viscosity [81]; therefore, it was considered as a potential drilling fluid in the next series of experiments.

Gel strength characterizes the ability of the flushing fluid to hold suspended particles of the rock and gas bubbles, as well as to penetrate cracks and pores of rocks and hold there under the influence of loads. The value of gel strength is crucial for understanding the thixotropic properties of a liquid that characterize its ability to reduce viscosity from mechanical stress and increase viscosity at rest. The measurements provided results in 1 min: initial torsion angle  $\varphi$  was 20°, torsion angle after the stress  $\Delta \varphi$  was 204°; and in 10 min: initial  $\varphi$ —102°,  $\Delta \varphi$  after the stress—49°. Gel strength (Pa) is calculated as  $\theta = \mathbf{k} \times \Delta \varphi$ , where *k* is the gel strength coefficient at the 1° torsion, mg/cm<sup>2</sup>; k is a constant for a given wire elasticity, which in turn depends on the thickness of the wire and is equal to 0.450 mg/cm<sup>2</sup> at the 0.3 mm diameter. The calculated values are  $\theta_1 = 0.450 \times 204 = 91.80$  Pa and  $\theta_2 = 0.450 \times 49 =$ 22.05 Pa. Thixotropic properties are optimal at the coefficient  $K_t$  ( $\theta_1/\theta_2$ ) equal to 1.0–1.5. The calculated coefficient is K<sub>t</sub> = 91.80/22.05 = 4.16 that indicates the need for certain measures to make the thixotropic properties of the clay suspension more suitable.

The fluid loss of the drilling mud is the ability to filter out the liquid phase under the influence of excess pressure. It is the supreme significant parameter when drilling in loose, porous, and fractured rocks as clay, mud with high fluid loss forms a thick and friable crust, narrowing the wellbore and causing tightening and sticking of the drilling tool when lifting. The inflow of water filtrate into rocks surrounding the wellbore can cause swelling and collapse of the rocks, as well as a decrease in the production rate. Fluid loss reduction can prevent these complications [82]. The obtained average value of water loss amounted to 24.25 cm<sup>3</sup> in 30 min (Table 6). This value is standard for clay mud. For normal clay solutions, fluid loss is considered permissible if its value does not exceed 10–25 cm<sup>3</sup> in 30 min. When drilling in challenging conditions of loose or unstable rocks, the fluid yield of the solution is to be reduced to 5 cm<sup>3</sup> in 30 min or less. The thickness of the clay cake on the filter was 1.5 mm. It is also within acceptable boundaries, which are 1–2 mm.

| Sample Series               | DF-1 | DF-2 | DF-3 | DF-4 |
|-----------------------------|------|------|------|------|
| Fluid loss, cm <sup>3</sup> | 24.0 | 25.0 | 23.5 | 24.5 |

Table 6. The leakage rate of the liquid phase of the drilling fluid mixture series (DF) over 30 min.

To reduce fluid loss, 15–30% sodium silicate or up to 25% of various sodium and potassium salts (customarily NaCl or KCl) are added to the clay solution. To stabilize the solution, up to 2% carboxymethyl cellulose (CMC or cellulose gum) and liquid silica (up to 50%) can be added [83,84]. At this time, liquid glass is practically not used in drilling fluids, being replaced by modern polymeric materials based on modifications of hydrolyzed polyacrylonitrile, polyacrylamide, etc. Carboxymethyl cellulose with various degrees of polymerization is used in drilling fluids in the present day.

Based on the obtained indicators, it is possible to recommend the suspension of the pond zone purified from large impurities as a drilling fluid. However, its practical use as such is limited by several factors, such as its increased swelling.

## 4. Conclusions

Three directions of saponite clay reutilization were studied in the conditions of Northwestern Russia: production of aqueous clay-based drilling fluid, cement clinker and ceramics manufacture, and integration of alkaline clay into the reclamation of acidic peat bogs. The empirically tested hypothesis on their suitability showed that the cold climate and the specific consumer market are of lesser importance than the technological constraints. Despite the high demand for drilling operations in the region, the saponite suspension will be marginally applicable due to the fluid loss of 24.25 cm<sup>3</sup> in 30 min that can cause swelling and collapse of the rocks, as well as a decrease in the production rate. The obtained samples of ceramics and cement clinker are of satisfactory strength: the average collapse pressure of 13.53 MPa at 800 °C; a positive facet of the building material manufacture is the opportunity of including bottom ash additive from the slag fields of coal thermal power plants (5–10 wt% at 1400–1500 °C) to reduce the moisture content of cement slurry and fuel consumption for clinker burning. Furthermore, tail grinding occurs during the preparation of the raw material for Portland cement; this waste reprocessing is of high importance for the use of clay pulp as a neutralizing agent, as fine particles are required for the reaction. Experiments on the Histosol deacidification with the alkaline clay slurry demonstrated that neutralization with the average pH level of 7.1 is reached at 30% of the pulp added and an experimental site with perennial grasses proved the efficacy of the technique. Moreover, the reclamation of disturbed lands is an integral part of the social and environmental responsibility of the mining company and this scenario addresses the community necessities at both local and regional levels. In summary, (i) kimberlite processing waste can be applied most beneficially as a peat soil amender; (ii) production of construction materials is technically possible, although, the demand is volatile in the underpopulated area; (iii) drilling mud is stably required by the provincial mining industry but the saponite-based liquid requires extra spending on additives to reduce fluid loss.

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