recycling

MDPI

Article

Development of a Recycling Process for the Recovery of Gypsum Stone from Stockpile Material †

Jacob Fenner *, Julius Luh, Bengi Yagmurlu and Daniel Goldmann

Department of Resource Processing Technologies, Institute of Geotechnology and Mineral Resources, Clausthal University of Technology, 38678 Clausthal-Zellerfeld, Germany; julius.luh@tu-clausthal.de (J.L.); bengi.yagmurlu@tu-clausthal.de (B.Y.); daniel.goldmann@tu-clausthal.de (D.G.)

* Correspondence: jacob.fenner@tu-clausthal.de

† This article is a revised and expanded version of a paper entitled "Entwicklung eines Recyclingverfahrens zur Minimierung und Verwertung von Rückständen aus der Gipsgewinnung und-aufbereitung", which was presented at "Berliner Konferenz für mineralische Nebenprodukte und Abfälle", Berlin, on 26 April 2022.

Abstract: Due to changes in the German government's energy concept, the amount of gypsum produced in flue gas desulfurisation plants (FGD gypsum) will fall from 5 million tons per year to 1 million tons or less by 2038 at the latest. As of 2016, FGD gypsum accounts for 55% of German gypsum mix. The resulting raw material gap must be closed through innovative recycling concepts, such as the processing of existing mine dumps. The process development aims to achieve a calcium sulfate dihydrate content of 85% and a reduction in the stockpile volume by 50%. The main components of the stockpiles are calcium sulfate in the form of gypsum stone as well as clay minerals and organic matter. Successful laboratory tests were transferred to a pilot scale jigging machine with dewatering screening. The process water is circulated throughout the entire process. The gypsum content in the heavy fraction is 76% when measured with ICP OES and 87% when measured via thermogravimetric methods. Furthermore, pilot-scale dry screening on the stockpile took place, and up to 1500 tons of material could be processed. Due to fluctuating weather conditions, the screening quality was subject to significant variations. Under optimal conditions, up to 60% of the feed could be recovered as gypsum stone; however, the screening process was nearly impossible during rain; therefore, a process combination of screening and a downstream jigging machine is recommended.

Keywords: gypsum; jigging machine; stockpile; tailings recycling; process development; pilot scale

check for updates

Academic Editors: Sossio Fabio Graziano, Rossana Bellopede, Giovanna Antonella Dino and Nicola Careddu

Received: 14 November 2024
Revised: 10 January 2025
Accepted: 13 January 2025
Published: 16 January 2025

Citation: Fenner, J.; Luh, J.; Yagmurlu, B.; Goldmann, D. Development of a Recycling Process for the Recovery of Gypsum Stone from Stockpile Material. *Recycling* **2025**, *10*, 12. https://doi.org/10.3390/recycling10010012

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/).

1. Introduction

Adopting the German government's energy concept, planned in 2010 and submitted in 2020, laid the foundation for a shift in German energy policy [1,2]. As part of the energy concept, the German government plans to phase out coal by 2038 at the latest, with the aim of bringing this forward to 2030 [3]. The planned phase-out of coal-fired power generation will largely eliminate the supply of gypsum from flue gas desulfurisation plants (FGD gypsum) in its current form [4,5]. It is expected that the amount of FGD gypsum currently produced will decline from around 5 million tons to 1 million tons or less by 2038 at the latest [2,4,6,7].

Gypsum, known as calcium sulfate dihydrate, can occur naturally or be produced through industrial processes [3,8,9]. Gypsum is mainly produced by the chemical reaction of sulfur dioxide (SO_2) with limestone (calcium carbonate, $CaCO_3$) in a process known

as flue gas desulphurization. This reaction takes place in FGD plants installed in coal-fired power plants. In this process, the sulfur dioxide released during the combustion of coal is treated with limestone, resulting in the formation of gypsum (calcium sulfate dihydrate, $Ca[SO_4]$-$2H_2O$) [10,11]. There are also natural deposits of gypsum formed by geological processes over millions of years. These natural gypsum deposits are often found in the form of gypsum stone or anhydrite ($Ca[SO_4]$), whereby anhydrite is formed when water is extracted from gypsum [12,13]. With its physical properties, such as a density of 2.3 to 2.8 g/cm^3 and hardness on a Mohs scale of 2 [10,14], it is considered an important material for modern and flexible construction solutions for walls, ceilings, and floors in residential and commercial buildings. By separating load-bearing, durable shell construction from contemporary interior finishing, gypsum allows for individual adaptation and design of the existing building fabric [8,15]. Despite being widely used in the construction industry, gypsum is also used in agriculture, the pharmaceutical sector, and for arts and decoration [10]. Further applications for gypsum are in the food industry as E 516 [16], animal beddings [16], and environmental applications such as wastewater treatment [11].

From 2004 to 2016, Germany extracted approximately 560 million tons of non-energy, mineral primary raw materials (excluding natural gypsum and anhydrite) annually. Additionally, around 102 million tons of primary raw materials were substituted by secondary raw materials in 2016. The extraction volume of natural gypsum and anhydrite amounted to 4.7 million tons in 2016 [6,13]. The Harz Region is one of the most significant regions for gypsum extraction. The Harz Region achieved a total revenue of nearly 340.7 million € in 2020, with 1057 employees directly involved in the industry [9]. Furthermore, flue gas desulfurisation plants produced about 6.5 million tons of FGD gypsum.

With the current gypsum mix in Germany consisting of about 45% natural gypsum, 55% FGD gypsum, and less than 5% recycled gypsum (RC gypsum), the resulting FGD gypsum significantly contributes to conserving natural gypsum deposits and preserving valuable ecosystems [6,7,17].

Considering the positive economic situation for the construction industry in recent years, it can be assumed that, compared with 2016, between 70% and 100% more natural gypsum and anhydrite will need to be mined or extracted through RC gypsum by 2035 [6,13]. This corresponds to about 3.6 to 4.5 million tons per year. Of the 214.6 million tons of mineral construction waste generated in 2016, only 0.3% (0.6 million tons) consisted of gypsum-based construction waste suitable for RC gypsum [7,18]. Since only 5% of this could be recycled, the loss of significant amounts of FGD gypsum cannot be covered by RC gypsum alone [6,7].

The disposal of products containing gypsum also poses a general problem when it comes to increasing recyclate quotas. In Germany, this takes place under the waste code numbers (AVV) 170801 (gypsum-based building materials contaminated with hazardous substances) and 170802 (gypsum-based building materials, with the exception of those under 170801) [4,5]. These enable the sorted collection of waste containing gypsum in the building materials sector; however, the composition and recyclability of waste containing gypsum varies greatly [19]. The introduction of additional AVV numbers for gypsum could help to increase the recycling rate and optimize waste management [12]. This would be both ecologically and economically beneficial [4,12].

The main contaminants in gypsum-containing waste can vary depending on the source and type; however, the most common are mineral impurities such as aerated concrete and screed [20], metallic impurities such as nails and screws [20], organic components such as paper, cardboard, and plastics and insulating materials [20] as well as general foreign

matter from other construction waste [12]. To achieve recyclability, the gypsum industry strives for an impurity content of less than 5% [20].

Current research approaches to increase recycling rates often focus on the recycling of gypsum plasterboard [14,21], recycling with subsequent use of the RC gypsum as construction material in general [22], in specific concrete mixes as a cement substitute [16] or as a bloating component in lightweight aggregates [23,24]. In addition, several LCAs are comparing FGD gypsum projects [25,26] to optimize the temperature range and calcination times in the thermal part of the recycling process [27] and natural gypsum as well as projects on closing material cycles [11,28] and reducing the environmental impact of landfilling [16]. Stockpile recycling mainly focuses on phosphogypsum [29,30]. Primary gypsum mining in the Harz Region usually comes with compact and homogeneous rock deposits. There is barely any research about recycling stockpiles of non-homogenous primary gypsum deposits [31,32].

This research aims to characterize the processing residues generated by the industrial partners, optimize the current processing methods, and develop a new pilot process to make the stockpiles reusable in production. The outcomes of this research could also be applied to other tailings piles in the region. By implementing appropriate crushing, classification, and both dry and wet sorting steps, a process is developed to separate mineral impurities from the gypsum rock, increase the recyclable fraction to achieve a gypsum content of 85%, and reduce the stockpile volume by 50%. In this context, "gypsum content" and "calcium sulfate dihydrate content" are used interchangeably.

2. Results

The experimental plan includes preliminary screenings, laboratory tests, and pilot plant tests. The required reduction in volume of the existing stockpiles by 50% compared with the total mass relinquished is expressed through the mass recovery (R_m). Parts of the results have already been presented at the "Berliner Konferenz für mineralische Nebenprodukte und Abfälle" [33]; further results were added for this article.

2.1. Preliminary Observations

Initial preliminary tests revealed that, regarding lab-scale screening, the stockpile material was nearly impossible to screen in its muddy state without sufficient process water, as the screen bottoms tended to be clogged. Adding water allowed clean gypsum stones to be obtained for analysis in the screening tower.

A linear vibrating screen with a 4 mm wire mesh and a drum screen with a 10 mm square mesh were used. In both screening tests, clay and loam adhesions remained on the gypsum stone, while organic matter contained in the material was discharged with the gypsum stone into the coarse material fraction. Loose clay and loam agglomerations are not adequately broken down by the spraying and mechanical stress of the screening machines. They are also discharged with the gypsum stone; therefore, wet screening on continuously operating machines is not successful.

Preliminary tests in batch laboratory drum mills (with a drum volume of approximately 4 L) were also unsuccessful, both with completely dried and wet material. Due to prior drying, the previously moist agglomerations on the gypsum stone solidified. The comminution energy introduced by autogenous grinding could not break apart the agglomerates. Repeating the test with moist material and additional washing water, in this case using the grinding drum as a lautering drum, was also insufficient in terms of results, even though compared with wet sieving, the free agglomerates and adhesions were better moistened and wetted.

2.2. Laboratory Scale Trials

2.2.1. Impact Mill

Impact mills are suitable devices for the fine comminution of particles ranging from 0.05 mm to 10 mm. They are particularly effective for brittle and hard materials, such as combining gypsum stone and clay agglomerations, where pressure and impact stress are efficient comminution methods [34]. The impact mill used in this study is a rotor impact mill featuring a grinding chamber designed as an annular space, a rotor circumference of 0.27 m, fixed impact bars, and two spring-mounted impact plates arranged in series. Rotor impact mills typically operate at high speeds, with rotor circumferential speeds between 100 and 150 m/s [35,36]; however, since the goal of the process is not to finely grind the feed material but merely to break off the agglomerations, peripheral speeds of 6 m/s to 18 m/s are used.

After the impact stress, gypsum stone separation occurs with a sieve cut of 1 mm, which could also be realized at a pilot plant scale. The mass recovery of the fraction greater than 1 mm peaked at a circumferential speed of 6 m/s and decreased with further increases in speed due to the higher energy input. Increasing the peripheral speed raised the proportion of gypsum particles found in the fines and increased gypsum loss due to dust formation. Analysis of the fraction greater than 1 mm reveals that circumferential speed does not significantly influence the gypsum content, which remained around 78%; however, the increased fine content resulted in a slight rise in gypsum content in the fraction less than 1 mm as circumferential speeds increased. A circumferential speed of 14 m/s was selected for subsequent tests, balancing mass output and gypsum content.

The complete drying of the stockpile material required significant energy, depending on the weather conditions; therefore, additional tests were conducted to observe how the material behaves at varying residual moisture levels. For comparison, a completely dried sample and samples with graded moisture contents up to approximately 5% were examined. To prevent post-drying during testing and analysis, the samples were crushed immediately at circumferential speeds of 14 m/s after removal from the dryer and were sieved directly afterward. As the residual moisture content increased, the gypsum content in the fraction greater than 1 mm noticeably decreased, while the gypsum content in the fraction less than 1 mm increased slightly. Residual moisture caused re-agglomeration of the gypsum stones post-crushing, resulting in an inaccurate screen cut and a decline in gypsum content in the fraction greater than 1 mm. As a result, while the selected screen cut allowed for a target mass recovery of 50%, the required quality standards were not met.

2.2.2. Jigging Machine

In the jigging process, fluid flows in pulses through the feed material placed on a jigging material carrier. During this process, the material is lifted by the upward flow and then settles back onto the carrier. This sedimentation leads to density-dependent rearrangement, enabling the separation of particles. As is typical in density-based processes, sorting by density can be influenced by classification effects [37,38].

A piston jigging machine with a continuously adjustable, sieve-like material carrier (0.8 mm mesh size) is preferred for laboratory tests. To ensure a harmonious jigging process, both the stroke height and the number of strokes must be coordinated to guarantee sufficient movement, even for coarse grain sizes [39,40]. The flow rate of the process water is kept constant throughout the jigging process.

The process duration varies in subsequent trials. The gypsum stone that remained as jigging material is free of clay residues after visual evaluation and appeared clean, as shown in Figure 1. With the previously established separation limit of 1 mm, the mass recovery in the jigging material fraction > 1 mm does not exceed 52%, regardless of the duration.

With longer process durations, the proportion of the fraction < 1 mm in the jigging material decreased slightly. The gypsum contents, with a maximum of 79%, are also below the process target of 85% and are similar to the samples obtained from the impact mill.



Figure 1. Jig screen plate with load, after 10 min process time.

2.2.3. Outcomes of the Laboratory Trials

Corresponding sieve content analyses can be derived from data generated in the laboratory analyses. For comparison, the sieve residue of each fraction, starting from the coarsest grain, is considered the discharged fraction and is evaluated in terms of its gypsum content (Figure 2). The larger fractions are always included in subsequent analyses of smaller grain sizes. While this method does not yet provide information about the mass recovery, it enables comparing the grain size-dependent gypsum content across the individual test series with the corresponding sieve sections.

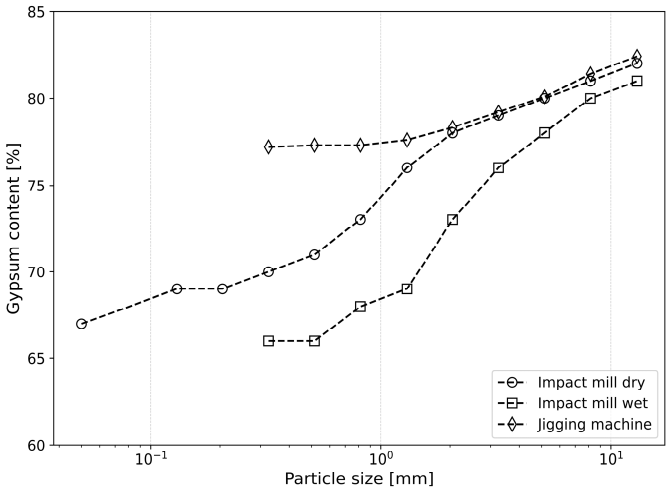


Figure 2. Comparison of gypsum contents from particle size analyses after impact crushing and jigging process.

The deviations in the valuable material content become evident when plotting the gypsum contents from the tests conducted with the impact mill and the jigging machine against grain size. For illustration, only the completely dried and undried samples are shown for the tests with moisture gradients in Figure 2.

To facilitate evaluation, the samples produced by impact crushing can be categorized into two ranges: larger and smaller than 1 mm. With completely dried material, the gypsum content of the samples crushed in the impact mill remained approximately consistent with

the gypsum content of the washed fractions from the jigging machine up to about 4 mm; however, in the fractions smaller than 4 mm, the proportion of remaining impurities—such as clay, loam, and organic matter—increased sharply, resulting in a decrease in recoverable gypsum content. In the previously defined second part of the screen cut (<1 mm), the gypsum content dropped to values between 65% and 70%, which corresponds to the gypsum content in the feed.

As the moisture levels increased, the gypsum contents of the fractions produced via the impact mill declined significantly. The gypsum contents of the completely dried samples outperformed those of the samples with residual moisture across all fractions. In the fractions greater than 10 mm and between 4 mm and 10 mm, the difference in valuable material content was primarily attributable to the qualities of the gypsum stone present. In contrast, moisture-related re-agglomerations caused by the contained clay dominated the fraction sizes smaller than 4 mm. The screening of the moist material below 0.4 mm is unfeasible, even with the use of screening assistants.

The rinsing water in the jigging machine suspends and subsequently discharges the existing agglomerates and impurities, resulting in a clean gypsum stone on the jigging tray. The organic matter present in the feed material is also suspended and carried away with the process water. Initially, the gypsum stone content of the fractions obtained from the jigging machine decreased uniformly by about five percentage points. After reaching the 1 mm mark, the content dropped only slightly, even with the addition of fractions smaller than 1 mm.

The process target of 85% gypsum stone content was not achieved with any investigated processes. The samples with residual moisture consistently fell below the results obtained with completely dried material. Based on the visual assessment of the results, it can be inferred that the decreasing gypsum stone contents in the jigging material, after removing impurities, solely depend on the quality of the contained stone itself. Consequently, the jigging process was selected for further upscaling to the pilot plant scale.

2.3. Pilot Scale Investigations

Although the target of the process was not reached completely, due to the positive results obtained in the laboratory tests, sorting stockpile material using jigging machines was pursued as the most promising method, and it was transferred to the pilot plant scale (see Figure 3). As the jigging process is quite complex and the legal requirements are higher, a dry screening process with a semi-mobile screening machine was tested on the stockpile.

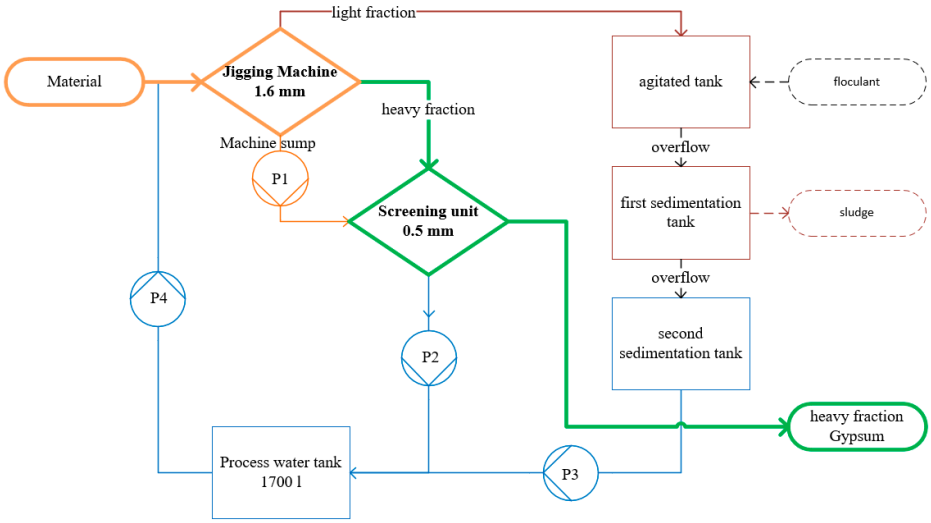


Figure 3. Process flow diagram of the pilot scale test.

2.3.1. Pilot Scale Jigging Machine

A vibrating jigging machine of the type SK 03-20 (see Figure 4) from SIEBTECHNIK GmbH was used for up-scaling tests. The available jigging surface measures 0.69 m^2, with a bed width of 300 mm and a bed length of 2300 mm. The polyurethane jigging bed used is slightly inclined, and features elongated holes along its entire length. Based on the laboratory tests, the required process water supply was determined as at least 60 m^3/h.

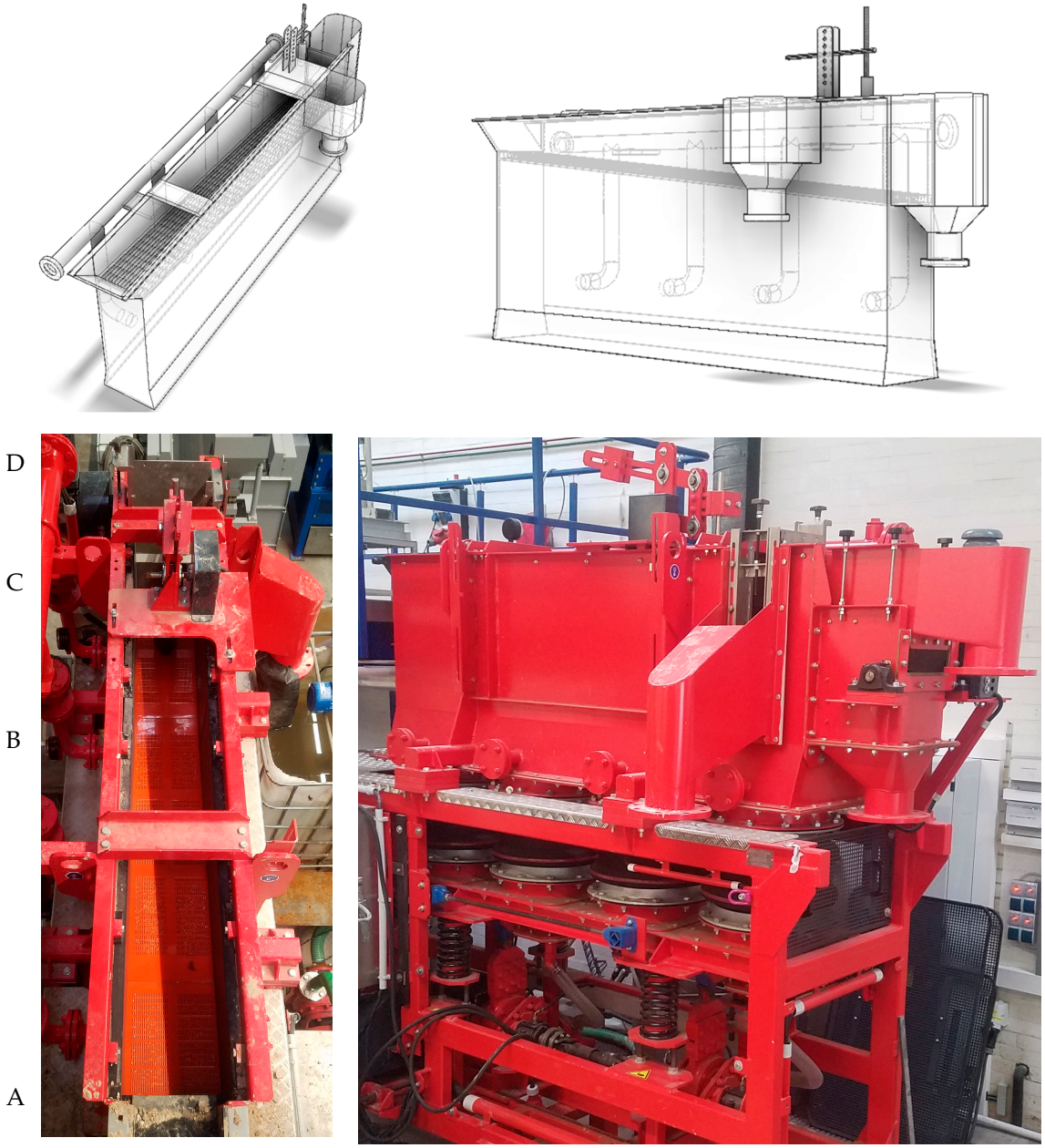


Figure 4. Oscillating jig type SK 03-20; feeding (A) with jig bed (B), conveying direction and discharge for light (C) and heavy material (D).

The parameters for stroke height and number of strokes were adjusted to 70 mm and 70 strokes per minute. Since it is impossible to supply fresh water in the required quantities, the process water is circulated, dissimilar to the laboratory scale tests in which a continuous flow of fresh water is used. Not circulating this amount of water is neither available nor sustainable for a pilot scale.

The process water from the heavy and light material discharges was combined in a storage tank. A centrifugal pump that was connected to the storage tank fed the process

water back to the jigging machine at a maximum rate of 75 m^3/h. Three IBC containers were connected in series on the light material side to protect the pump from excessive solid loads via an overflow and worked as sedimentation ponds. The first two IBCs primarily served as tanks to sediment solids discharged with the process water, while the third IBC worked as a buffer tank for feeding the process water back to the main storage tank with submersible sludge pumps.

The heavy material discharged at the front end was fed with the remaining process water to a downstream dewatering screen (PU fabric Lg = 0.5 mm) (Figure 5). No additional rinsing water was added for subsequent cleaning. While the process water was collected and returned to the process water circuit, the heavy material x > 0.5 mm could be separated without large amounts of residual water. Any particles < 0.5 mm in the heavy fraction were separated in a collection tray and reunited with the remaining heavy fraction after the end of the experiments.



Figure 5. Material discharge after dewatering screening.

The material is fed manually after start-up and regulation of the water circuit. The fed masses were logged, and the process water was sampled regularly for its solids content.

Approximately 700 kg of moist stockpile material were weighed for the pilot plant test described. After creating a stable water circuit and stable strokes, the material was continuously fed into the jigging machine. The gypsum stone discharged via the machine sump was included in the light material; however, since the corresponding discharges had to be removed from the process after a short time, the volume flow that the sump pump could convey is not considered further in the balance. With a test duration of 37 min and a constant opening of the heavy material barrier, a mass flow of just under 1 Mg/h of earth–moist stockpile material was achieved. The solids content pumped in the process water at the end of the test was 32 g/L.

In subsequent tests, the mass flow in the feed was increased to 3 Mg/h of ground-moist material. In follow-up tests, a successful reduction in the size of the jig carrier from 1.6 mm PU mesh via 1.2 mm steel mesh down to 0.8 mm PU mesh was also carried out. After finishing the experiment, about 360 kg of rock (dry mass) could be recovered as heavy material. To protect the plant, particularly large lumps of rock 200 mm or more were picked by hand parallel to the material feed. This fraction accounted for 19 kg of dry matter at the end of the trial. The 360 kg of heavy material recovered corresponds to a mass recovery of approximately 57%. The successful implementation thus met the process target of at least 50% mass output. About 40% of the feed material < 0.5 mm was spread with the process water and dewatering screening in the light material.

2.3.2. Screening Machine

As mentioned above, the jigging process is quite complex and comes with high legal requirements regarding water use and process water treatment. As the pilot plant tests confirmed, a protective screening of at least 200 mm mesh should be installed before the jigging process. This improvement was implemented by using a mobile screening machine of KLEEMANN (see Figure 6). In addition, it is investigated whether the fractions obtained during dry weather seasons can already be utilized without a jigging process.



Figure 6. Pilot plant for stockpile screening during operation.

During the installation of the screen, it became apparent, as shown in the lab tests before, that the weather and the stockpile quality strongly influenced the results of the protective screening. The fluctuations in quality were also evident in the gypsum content of the heavy material from the pilot plant tests on the jigging machine.

Rigid, hexagonally perforated screen panels were used in the upper deck, while a self-cleaning square mesh was employed in the lower deck. The tests indicated that the organic material tended to tilt upwards when it hit the screen and slipped through the meshes. Sliding and equalizing mats were installed in the upper quarter to counteract this. Sliding over the mats caused the organic materials to realign horizontally on the screen deck. Consequently, a significant portion of the organic matter, including roots and woody plants, was separated in the upper deck, alleviating the burden of organic impurities on subsequent processes (see Figure 7).



Figure 7. From left to right: Sieving without homogenizing mats, sieving with homogenizing mats, organics in the sieved oversized grain, organics as clamp grain in the upper deck.

By positioning the screen on top of the stockpile, the intermediate fraction could be discharged over the edge of the pile. When the material was sufficiently dry, the rolling on the edge of the stockpile facilitated the shearing and breaking of clay adhesions on the gypsum stone—resulting in rather clean gypsum rocks. This effect did not occur in the lab

tests due to the sheer size of the stockpile; however, it is important to emphasize that this effect depended highly on the weather conditions and required extended dry phases with strong sunlight. In contrast, rolling over the clay-containing discharge edge with moist material could lead to agglomeration, similar to the principle of a pelletizing plate.

This was reflected in the results (Table 1): when the material was completely dry, between 30% and 60% of the gypsum could be recovered by screening, depending on the stockpile quality. In rainy conditions or with wet material, this percentage dropped to between 20% and 50%. During rain, the clay content of the material made it almost impossible to screen, so only 20% was recovered in the stone fraction and 80% in the overburden.

Table 1. Output of pilot scale screening on site.

Material	Moisture Content	R_m Gypsum Rock	R_m Overburden
rock-rich	sun-dried	60%	40%
clay-rich	sun-dried	30%	70%
current process residues	sun-dried	30%	70%
rock-rich	earth–moist	50%	50%
clay-rich	earth–moist	20%	80%

3. Materials and Methods

For process delimitation and potential assessment, the stockpile material and the stockpile body are first characterized, and suitable analytical methods are selected and presented below.

3.1. Stockpile and Material Characterization

The stockpile bodies are first inspected with the industrial partner CASEA to characterize the existing stockpiles. This inspection reveals that the existing stockpile is divided into two bodies. In the left picture of Figure 8, the old stockpile in the top left corner (framed in red) can be seen; birch, pine, and spruce started growing on top, contaminating the material with organic matter, Figure 8—top right. Right next to the old stockpile in the top center of Figure 8—left, the second, younger stockpile can be seen. The younger stockpile is still actively used and devoid of vegetation. In the following analysis, only the older stockpile body no longer in use will be considered.



Figure 8. (**Left**): mining and production site—stockpile highlighted in red box, (**top right**): on top of the stockpile, (**bottom right**): the result of the protective screening of the stockpile material, gypsum stone 200 × 200 × 200 mm.

The stockpile has been in operation since 1950; at its widest point, the cone measures around 250 m and now has an estimated total tonnage of 1 million tonnes of material. Depending on the weather and rock quality, the screen cut in the associated production plant nowadays varies between 40 mm and 100 mm and is stored in the younger stockpile. Over the years, many optimizations have been made to the existing production line. Precise information on this is not available. It can, therefore, only be assumed that the material deposited on the analyzed stockpile is, on average, coarser-grained than that deposited on the newer stockpile.

Sampling is conducted throughout the entire body of the stockpile and is focused near the surface. The earth–moist samples are processed to remove particles larger than 32 mm using a protective sieve. The fraction above 32 mm can be considered pure gypsum in terms of primary particle size Figure 8—bottom right. The fractions below 32 mm are then divided to the analysis size using a riffle divider and wet sieved to obtain the particle size distribution shown in Figure 9. The R 5 sieve series, according to Renard was used for sieving; this is a simplification of the R 20 sieve series [34–36]. The mesh sizes used are 1.0 mm, 1.6 mm, 2.5 mm, 4 mm, 6.3 mm, and 10 mm. The series values can be multiplied by powers of ten as required.

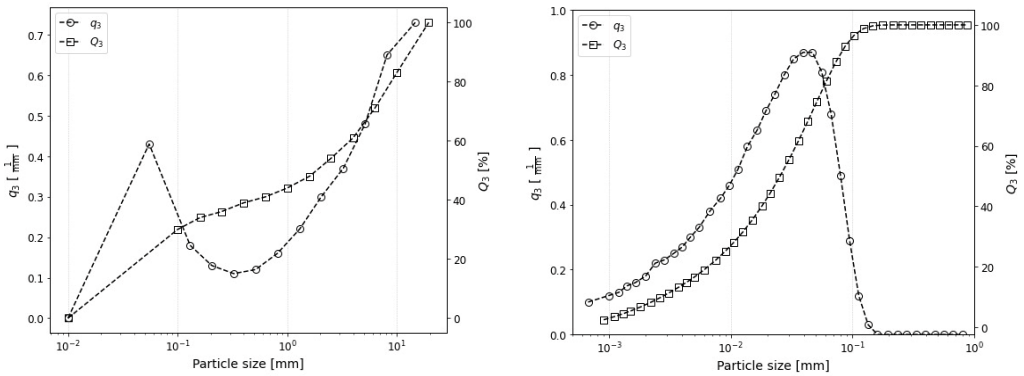


Figure 9. Particle size distribution of stockpile material after protective screening with 32 mm. (**left**) wet screening of particles below 32 mm, (**right**) Laser diffraction of particles below 100 µm.

The entire grain band exhibits significant fluctuations. The largest grains are rock lumps with edge lengths of up to 200 × 200 × 200 mm. The fraction produced by protective screening is not included in the following analysis as the particles are too large for lab-scale analyses and consist mostly of pure gypsum rock.

The particle size analysis of the tailings by screening (Nexopart, EML 200, Gersthofen, Germany) and laser diffraction (Sympatec, HELOS KR/QUIXEL, Clausthal-Zellerfeld, Germany), as shown in Figure 9, reveals two main fractions. The gypsum fraction, with grain sizes greater than 0.5 mm, dominates, accounting for 60% to 70% of the total sample. The remaining 30% to 40% is attributable to the clay fraction, which has grain sizes less than 0.5 mm containing mostly clay, gypsum sand from crushing and fine fillers. About 80% of the stockpile material is larger than 10 mm. The average grain size, which corresponds to 50% of the material, is 2 mm. The grain size class with the smallest proportion of particles, x_{min}, is between 0.4 and 0.6 mm.

The objective of the process is to reduce the volume of the stockpile by 50%, which would correspond to a screen cut of 2 mm. In the fractions greater than 0.63 mm, the dihydrate contents, determined by crystal water analysis, range from 84% to 87%, aligning with the 85% gypsum content process target; however, in the fractions smaller than 0.63 mm, the dihydrate contents drop to about 66%, and the clay particles increase. Larger woody

organic fractions and leaf residues can be separated during protective screening, while the remaining organic residue is distributed across all fractions.

X-ray diffraction measurements using powder with a PANalytical X'Pert-PRO diffractometer equipped with a Co X-Ray tube (Malvern Panalytical, Malvern, UK) can identify calcite and quartz as accompanying minerals in the fraction greater than 10 mm. In the fraction smaller than 1 mm, calcite, quartz, illite, and rutile are accompanying minerals. The fraction less than 0.1 mm also contains microcline, clinochlore, magnesiohornblende, sanidine, muscovite, and other potassium compounds that cannot be clearly identified. The dominant mineral phases, gypsum, and anhydrite, are found in all fractions.

3.2. Characterization Methods

There are numerous methods for determining calcium sulfate dihydrate. One widely used industrial method is determining gypsum content through the mass loss of expelled water of crystallization. Another option is measuring the total sulfur content of the sample using optical emission spectroscopy, followed by extrapolation to the calcium sulfate content.

3.2.1. Chemical Calcium Sulfate Determination

One possibility for analyzing the gypsum content is the determination of the sulfur content by optical emission spectroscopy with inductively coupled plasma (hereafter referred to as ICP-OES). For this purpose, the sample is ground to an analytical fineness of less than 0.1 mm, digested with aqua regia, and the total sulfur content is determined. The calcium sulfate content of the sample can be calculated from the sulfur content; however, the calcium sulfate content alone does not allow a statement on the hydration phases.

It is known that the site does not consist only of calcium sulfate dihydrate; therefore, it is assumed that 85% of the calculated calcium sulfate represents natural gypsum. The remaining 15% is accounted for by anhydrite and intermediate rock. This proportion is considered a factor in the evaluation when calculating the dihydrate content.

3.2.2. Thermal Calcium Sulfate Determination

Another method for determining the dihydrate content is via crystal water determination (CWD), a simplified form of thermogravimetry widely used in industry; therefore, the sample to be analyzed is ground to less than 0.5 mm and initially heated to 40 °C to remove the free moisture. The sample is then heated to 360 °C, where the water of crystallization contained in it is expelled, and the resulting loss of mass is calculated by the integrated load cell. If this mass loss corresponds to the water of crystallization expelled from calcium sulfate dihydrate, the sample's calcium sulfate dihydrate content can be calculated.

4. Discussion

Within the scope of this study, the gypsum content was determined solely by ICP-OES during preliminary and laboratory tests. Both analytical methods were used for comparative purposes to characterize the stockpile material and evaluate the pilot plant test. The total contents of calcium sulfate dihydrate determined by ICP-OES varied between 60% and 80%, depending on the sample. In contrast, repeating measurements via CWD led to total contents between 70% and 90% calcium sulfate dihydrate. The gypsum contents measured in the stockpile characterization using CWD were, on average, found to be about 5% to 10% higher.

However, it is important to note that due to the presence of the clay and the organic material, the indicated mass loss may be higher than the actual expelled crystal water content. Control measurements with pure calcium sulfate dihydrate showed a deviation of 2% compared with the defined initial content. When extrapolating to calcium sulfate

dihydrate, the error increased to an average of 5.5%. In samples standardized to 100% calcium sulfate dihydrate, concentrations of only 94.5% were determined. Nevertheless, CWD is the more common method in the industry. The process of analyzing is much simpler, cheaper, and faster.

Compared with the jigging machine and impact mill, lautering drums were also excluded in terms of the long process time in the drum. At least for the laboratory tests, the organic matter remained in the material, and additional dewatering screening was required. Finally, the disaggregation of the completely dried material through impact stress and the combination of washing and jigging processes in a laboratory jigging machine was examined in the context of the preliminary tests.

The principle of selective comminution of clay agglomerates and subsequent separation from the gypsum carrier rock has proven effective; however, it has been observed that comminution using an impact mill lacks sufficient selectivity, resulting in the concomitant comminution of significant amounts of gypsum rock. Consequently, the 'chipping off' of the clay-containing deposits was only partially effective, as in some instances, the agglomerates of gypsum and clay were completely comminuted, resulting in an increased proportion of fines being produced. Notably, in fractions smaller than 4 mm, there was a substantial increase in the content of clay particles; however, as soon as residual moisture was present in the material, the results rapidly deteriorated to gypsum contents of only 65%.

The quality of the material extracted from the jig also decreased the finer the screen cut was selected; however, as the clay fraction was gently washed out of the rock by the process water, the gypsum content stabilized between 75% and 80%. With the impact mill, the quality deteriorated continuously as the screen cut became finer.

The mass output of the pilot scale jig confirmed the results of the stockpile characterization shown in Figure 9, with a distribution of 60% gypsum rock and 40% clay fraction after dewatering. The gypsum content in the heavy fraction was 76% and 87% when measured by ICP-OES and CWD, respectively. The recovery rate of gypsum in the heavy fraction compared with the source material was approximately 60%.

By transferring the test to the pilot plant scale, the results of the characterization and laboratory tests were verified. In particular, recirculating the process water was challenging due to the small amount of water in the storage tank and the large amount of process water required for the task. As this was not the focus of the work, the process water treatment and recovery of solids from the process water is not described in this study.

Under favorable weather conditions, the screening machine yields results comparable to those of the jig, achieving an extraction rate of approximately 60% gypsum rock; however, in poorer-quality material or adverse weather conditions, the efficiency of gypsum extraction dropped significantly to around 30%. These findings indicate that the standalone use of the screening machine is only practical during optimal weather. Alternative or supplementary processing methods should be considered to maintain extraction efficiency and material recovery in less favorable conditions.

Although linking experimental results to the existing literature is critical, this study employed the machines in a novel and unconventional manner that has not been previously published. As a result, a comparison with existing studies is not possible/reasonable. The methodological innovation of this study highlights a gap in the literature. It underscores the need for further investigations to establish comparable datasets and validate these findings in similar experimental contexts.

5. Conclusions and Outlook

The tests have demonstrated that it is possible to process residues from primary gypsum extraction contaminated with clay using appropriate process combinations and

successfully transferring results from the laboratory to the pilot scale. The analysis of gypsum content—via sulfur content measurement using ICP-OES or thermogravimetric determination of crystal water—provided comparable results only to a limited extent. Further investigations are necessary to ascertain which method is more reliable. Thermogravimetric determination is preferable for practical applications because of its simplicity compared with the more complicated ICP-OES procedure. A potential solution, albeit very complex for continuous industrial processing, could involve combining both methods with quantitative X-ray diffraction (XRD) measurements.

The experiments revealed that even a residual moisture content of 2% in dry processing can lead to quality losses. Drying or storing the material in weather-protected storage areas requires significant energy, space, and time. By integrating dry screening, washing, and jigging processes, it is possible to process tailing material in a manner that is largely independent of weather conditions and gentle on the material, except during frost periods. The transfer of tests and parameters from the laboratory scale to the pilot plant scale has been conducted successfully.

It has been observed that short and fast strokes gain better results than long, slow strokes with more dwell times. By adapting the stroke frequency, further improvements can be achieved in the pilot plant. Dissimilar to classical jigging processes, there is no density separation in the actual sense when separating a light fraction. Instead, the movement of the process water and the material in the jigging bed creates gentle stress, leading to the disaggregation and dispersion of adhering clay particles. Dry deagglomeration through crushing tends to distribute organic matter—such as roots and leaves—across all fractions. In the jigging process, the organic matter is discharged with the clay fraction via process water into the light fraction, resulting in gypsum stone extracted in the heavy fraction being free of organic impurities.

Manual feeding and picking currently limit the mass throughput for pilot scale testing; however, the mass flow can easily be increased with appropriate conveying technology and protective screening. Additional rinsing water can be introduced after cleaning the dewatering screen to enhance the quality. To prevent clogging of the nozzles, either process water should be treated before recirculation or fresh water should be used.

Future process investigations should explore options for dewatering the light fraction and treating process water. Coarser components of the light fraction can be effectively separated through sedimentation without flocculation agents; therefore, the addition of flocculation agents may become unnecessary. Residues of fine-grained gypsum stone, organic matter, and clay in the light fraction can be utilized for the renaturation of the quarry. The results indicate that a stable process water cycle can be established even with relatively small amounts of fresh water, allowing the process to operate over extended periods with larger feed material quantities without excessive water consumption.

Parallel to the development of the jigging concept, screening tests conducted on the tailings stockpile have demonstrated considerable success. Throughout the test series carried out in the spring and summer of 2023 and 2024, more than 1500 tons of tailings material were processed through screening. The resulting oversize fraction is currently incorporated into the primary gypsum production process, provided that the material quality meets the required standards, dependent on dry and sunny weather conditions. The medium-sized fraction is supplied to a cement manufacturer as a cement regulator, while the undersized fraction remains on-site for cultivation measures. A combined process is planned to facilitate the direct utilization of dry-weather fractions and the extended processing of rainy-weather fractions through semi-mobile jigging machines provided by SIEBTECHNIK. While the current concept shows promise, further optimization is necessary to enhance its efficiency and applicability. The approach is transferable to other mines and

quarries in the region that share geologically similar characteristics. However, additional testing is required to evaluate its effectiveness across a broader range of regional mining stockpiles, potentially extending its applicability to more diverse geological conditions.

The research demonstrated that processing gypsum tailings with clay contaminations using a jigging machine is a viable and effective method. However, the results highlight that a protective screening is necessary. However, the results were satisfactory when the stockpiled material was processed with the tested screening machine under ideal conditions. The impact mill proved to be somewhat effective in removing adherent clay particles. Still, its suitability for this purpose remains limited as it also comminutes the target material and proved too susceptible to residual moisture in the material.

The implementation of the proposed recycling concept for the stockpile has the potential to make a substantial contribution to regional gypsum recycling efforts and the conservation of natural resources. This is particularly relevant for the Southern Harz region and the associated landscape conservation area 'Karstlandschaft Südharz,' which encompasses one of Germany's most valuable gypsum deposits. The area has become a focal point of tension between environmental protection and the need to secure regional raw material supplies [41], and recycling existing stockpiles is emerging as a key strategy. This is because it supports conservation efforts, contributes to the local raw material supply chain, and helps secure valuable employment opportunities in the region.

Author Contributions: Conceptualization J.F. and J.L.; methodology J.F.; validation J.F. and J.L.; experimental investigation J.F.; data curation J.F.; original draft preparation J.F. and J.L.; review and editing: J.F., J.L., D.G. and B.Y.; visualization J.F., J.L. and B.Y.; supervision D.G. and B.Y.; project administration D.G. and J.F.; funding acquisition D.G. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded within the framework of the research project "TreSor—Gips" by the Federal Ministry of Education and Research (BMBF) of the Federal Republic of Germany in the context of the framework of the funding guideline "WIR! Wandel durch Innovation in der Region" (FKZ 03WIR0305B).

Data Availability Statement: The data presented in this study are available from the corresponding author upon reasonable interest.

Acknowledgments: The authors would like to express their gratitude to the TreSor-Gips research project for the financial support. We would also like to thank the institute's analytical laboratory for the great teamwork. We acknowledge support from the Open Access Publishing Fund of Clausthal University of Technology.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Bundesministerium für Wirtschaft und Klimaschutz. Kohleausstieg und Strukturwandel. Available online: https://www.bmwk.de/Redaktion/DE/Artikel/Wirtschaft/kohleausstieg-und-strukturwandel.html (accessed on 18 December 2024).
2. Demmich, J.; Schmitt, H. Knauf KG Der Rohstoff Gips im Wandel der Zeit: Unter dem Gesichtspunkt Erneuerbarer Energien; in 3. BGR-Rohstoffkonferenz: Hannover, Germany, November 2017. Available online: https://www.bgr.bund.de/DE/Themen/Min_rohstoffe/Veranstaltungen/Rohstoffkonferenz2017/Demmich.pdf?__blob=publicationFile&v=2 (accessed on 18 December 2024).
3. Lapp, M.; Ganswind, F.; Günther-Plönes, A.; Ehling, A.; Fischer, H.-B.; Fox, O.; Schallschmidt, F.; Vulpius, B.; Dumperth, C.; Pustal, I.; et al. Gestein des Jahres 2022. Unternehmerverband Mineralische Baustoffe (UVMB) e.V. July 2022. Available online: https://www.geopark-thueringen.de/fileadmin/user_upload/Dateien/Galerie_Gesteine/Gestein_des_Jahres/2022_GdJ_Gips_Broschuere_web.pdf (accessed on 17 December 2024).
4. Alwast, H.; Bund für Umwelt und Naturschutz Deutschland e.V.—BUND. *Umweltverträgliche Alternativen zum Abbau von Naturgips*; Bund für Umwelt und Naturschutz Deutschland e.V.—BUND: Berlin, Germany, 2020. Available online: https://www.bund.net/fileadmin/user_upload_bund/publikationen/naturschutz/naturschutz_gipsgutachten.pdf (accessed on 18 December 2024).

5. Buchert, D.M.; Sutter, J.; Alwast, H.; Schütz, N. Ökobilanzielle Betrachtung des Recyclings von Gipskartonplatten. Available online: https://www.umweltbundesamt.de/sites/default/files/medien/1410/publikationen/2017-04-24_texte_33-2017_gipsrecycling.pdf (accessed on 18 December 2024).
6. Basten, M.; Bundesverband Baustoffe—Steine und Erden, e.V. *Die Nachfrage Nach Primär- und Sekundärrohstoffen der Steine-und-Erden-Industrie bis 2035 in Deutschland*; Bundesverband Baustoffe—Steine und Erden e.V.: Berlin, Germany, 2019. Available online: https://www.baustoffindustrie.de/fileadmin/user_upload/bbs/Dateien/Downloadarchiv/Rohstoffe/Rohstoffstudie_2019.pdf (accessed on 7 March 2022).
7. Schäfer, B.; Bundesverband Baustoffe—Steine und Erden e., V. *Mineralische Bauabfälle Monitoring 2016: Bericht zum Aufkommen und zum Verbleib Mineralischer Bauabfälle im Jahr 2016*; Bundesverband Baustoffe—Steine und Erden e.V.: Berlin, Germany, 2018. Available online: https://www.baustoffindustrie.de/fileadmin/user_upload/bbs/Dateien/Downloadarchiv/Rohstoffe/11_KWB_Monitoring-Bericht.pdf (accessed on 7 March 2022).
8. Bundesverband der Gipsindustrie e., V. *GIPS-Datenbuch*; Bundesverband der Gipsindustrie e.V.: Berlin, Germany, 2013. Available online: http://www.gips.de/fileadmin/user_upload/gipsdatenbuch_2013.pdf (accessed on 18 December 2024).
9. Mecke, M. Die Volkswirtschaftliche Bedeutung der Gipsindustrie im Harz, Aktualisierte und Erweiterte Regionalökonomische Analyse. DIW Econ GmbH. 3 November 2021. Available online: https://diw-econ.de/wp-content/uploads/DIW-Econ_Gipsindustrie-im-Harz_update-2021_v2.0.pdf (accessed on 18 December 2024).
10. Bustillo Revuelta, M. *Construction Materials: Geology, Production and Applications*; Springer Textbooks in Earth Sciences, Geography and Environment; Springer International Publishing: Cham, Switzerland, 2021; ISBN 978-3-030-65206-7.
11. Jiang, Z.-Y.; Sun, X.-P.; Luo, Y.-Q.; Fu, X.-L.; Xu, A.; Bi, Y.-Z. Recycling, reusing and environmental safety of industrial by-product gypsum in construction and building materials. *Constr. Build. Mater.* **2024**, *432*, 136609. [CrossRef]
12. Glodek, L.; Hoffmann, F.; Kess, M.; Sahner, J.; Schuster, R. GIPS Rohstoff und Lebensraum. GRÜNE LIGA e.V. January 2022. Available online: https://grueneliga.de/images/gips-broschuere_web.pdf (accessed on 18 December 2024).
13. Basten, M. *Die Nachfrage nach Primär- und Sekundärrohstoffen der Steine-und-Erden-Industrie bis 2040 in Deutschland*; Bundesverband Baustoffe—Steine und Erden e.V.: Berlin, Germany, 2022. Available online: https://cdn.prod.website-files.com/664355396b105bd9a4e9cadb/665dd3a096315c0e03fb96fd_2022-04-20_BBS_Rohstoffstudie_01_ONLINE.pdf (accessed on 18 December 2024).
14. Fedoskina, O.V.; Yu Svietkina, O.; Ziborov, K.A.; Yerisov, M.M.; Fedoskin, V.O. Gypsum recycling using an inclined chamber vibrating jaw crusher. *IOP Conf. Ser. Earth Environ. Sci.* **2024**, *1415*, 012126. [CrossRef]
15. Radeloff, D. *Herstellung und Entsorgung von Gipsplatten*; Stand: September 2007. in Umwelt-Spezial; Bayerisches Landesamt für Umwelt: Augsburg, Germany, 2007; ISBN 978-3-940009-39-5. Available online: http://www.bestellen.bayern.de/shoplink/lfu_abfall_00147.htm (accessed on 18 December 2024).
16. Hansen, S.; Sadeghian, P. Recycled gypsum powder from waste drywalls combined with fly ash for partial cement replacement in concrete. *J. Clean. Prod.* **2020**, *274*, 122785. [CrossRef]
17. Bastian, D.; Brandenburg, T.; Buchholz, P.; Huy, D.; Liedtke, M.; Schmidt, M.; Sievers, H. DERA Rohstoffinformationen: DERA—Rohstoffliste 2019. January 2019. Available online: https://www.deutsche-rohstoffagentur.de/DE/Gemeinsames/Produkte/Downloads/DERA_Rohstoffinformationen/rohstoffinformationen-40.pdf?__blob=publicationFile (accessed on 18 December 2024).
18. Bundesverband Baustoffe—Steine und Erden, e.V. *Mineralische Bauabfälle Monitoring 2020: Bericht zum Aufkommen und Verbleib mineralischer Bauabfälle im Jahr 2020*; Bundesverband Baustoffe—Steine und Erden e.V.: Berlin, Germany, 2023.
19. Gips Factsheet. Umweltbundesamt, Dessau-Roßlau, Projekt Factsheet. May 2019. Available online: https://www.umweltbundesamt.de/sites/default/files/medien/3521/dokumente/factsheet_gips_fi_barrierefrei.pdf (accessed on 18 December 2024).
20. Harder, J. Gips—Ein Knapper Rohstoff? Mineral Process. 2022. Available online: https://www.at-minerals.com/de/artikel/at_gips_ein_knapper_rohstoff_-3752852.html (accessed on 18 December 2024).
21. Weimann, K.; Adam, C.; Buchert, M.; Sutter, J. Environmental Evaluation of Gypsum Plasterboard Recycling. *Minerals* **2021**, *11*, 101. [CrossRef]
22. Boccarusso, L.; Durante, M.; Iucolano, F.; Langella, A.; Capece Minutolo, F.M.; Mocerino, D. Recyclability Process of Standard and Foamed Gypsum. *Procedia Manuf.* **2020**, *47*, 743–748. [CrossRef]
23. Fenner, J.; Zeller, A.; Liebezeit, S.; Knorr, M.; Schnell, A.; Mettke, L.; Goldmann, D. Investigation of Industrial Residues and Waste Materials to Expand the Raw Material Base for the Production of Lightweight Aggregates. *Mater. Proc.* **2024**, *15*, 71. [CrossRef]
24. Schnell, A.; Rübner, K.; Seher, J.; Müller, C.; Müller, A.; Liebezeit, S.; Fenner, J.; Martin, F.; Pniok, N. Manufacturing and Application of Lightweight Aggregates from Construction and Demolition Waste. *Chem. Ing. Tech.* **2024**, *96*, 969–975. [CrossRef]
25. Jimenez Rivero, A.; De Guzman Baez, A.; Garcia-Navarro, J. Gypsum Waste: Differences across Ten European Countries. *Int. J. Sustain. Policy Pract.* **2015**, *11*, 1–9. [CrossRef]
26. Jiménez-Rivero, A.; García-Navarro, J. Best practices for the management of end-of-life gypsum in a circular economy. *J. Clean. Prod.* **2017**, *167*, 1335–1344. [CrossRef]

27. Bumanis, G.; Zorica, J.; Korjakins, A.; Bajare, D. Processing of Gypsum Construction and Demolition Waste and Properties of Secondary Gypsum Binder. *Recycling* **2022**, *7*, 30. [CrossRef]
28. Jiménez Rivero, A.; Sathre, R.; García Navarro, J. Life cycle energy and material flow implications of gypsum plasterboard recycling in the European Union. *Resour. Conserv. Recycl.* **2016**, *108*, 171–181. [CrossRef]
29. Men, J.; Li, Y.; Cheng, P.; Zhang, Z. Recycling phosphogypsum in road construction materials and associated environmental considerations: A review. *Heliyon* **2022**, *8*, e11518. [CrossRef]
30. Murali, G.; Azab, M. Recent research in utilization of phosphogypsum as building materials: Review. *J. Mater. Res. Technol.* **2023**, *25*, 960–987. [CrossRef]
31. Fenner, J.; Goldmann, D. *Entwicklung eines Recyclingverfahrens zur Minimierung und Verwertung von Rückständen aus der Gipsgewinnung und Aufbereitung—Abschlussbericht*; Institut für Aufbereitung, Recycling und Kreislaufwirtschaftssysteme: Clausthal-Zellerfeld, Germany, 2024.
32. Losch, G.; TU Clausthal; CASEA GmbH. *Entwicklung eines Recyclingverfahrens zur Minimierung und Verwertung von Rückständen aus der Gipsgewinnung und Aufbereitung: TP 2: Charakterisierung und Aufbereitung*; Projektantrag: Ellrich, Germany, 2020.
33. Fenner, J.; Gaun, A.; Luh, J. Entwicklung eines Recyclingverfahrens zur minimierung und Verwertung von Rückständen aus der Gipsgewinnung und-aufbereitung. In *Proceedings of the Mineralische Nebenprodukte und Abfälle, Band 9—TK Verlag*; TK—Verlag: Berlin, Germany, 2022; Volume 9, pp. 144–157. Available online: https://books.vivis.de/produkt/mna9/ (accessed on 18 December 2024).
34. *DIN 66165-2:2016-08*; Partikelgrößenanalyse_-Siebanalyse_—Teil_2: Durchführung. Beuth Verlag GmbH: Berlin, Germany, 2016. [CrossRef]
35. *DIN 66165-1*; Partikelgrößenanalyse—Siebanalyse—Teil 1: Grundlagen. Beuth Verlag GmbH: Berlin, Germany, 2022. [CrossRef]
36. *DIN ISO 3310-1:2017-11*; Analysensiebe_—Technische Anforderungen und Prüfung_—Teil_1: Analysensiebe mit Metalldrahtgewebe (ISO_3310-1:2016). Beuth Verlag GmbH: Berlin, Germany, 2017. [CrossRef]
37. Schubert, H. *Mechanische Verfahrenstechnik*; Verfahrenstechnik; 3., erw. und durchges. Aufl.; Dt. Verl. für Grundstoffindustrie: Leipzig, Germany, 1990; ISBN 3-342-00381-2.
38. Wills, B.A.; Finch, J.A. *Wills' Mineral Processing Technology: An Introduction to the Practical Aspects of Ore Treatment and Mineral Recovery*, 8th ed.; Elsevier: Amsterdam, The Netherland, 2016; ISBN 978-0-08-097053-0. Available online: http://search.ebscohost.com/login.aspx?direct=true&scope=site&db=nlebk&AN=1060332 (accessed on 7 March 2022)
39. Pahl, M.H.; Brundiek, H. *Zerkleinerungstechnik*; Praxiswissen Verfahrenstechnik Mechanische Verfahrenstechnik; 2. Aufl.; Fachbuchverl.; Verl. TÜV Rheinland: Leipzig/Köln, Germany, 1993; ISBN 3-343-00843-5.
40. Schubert, H. (Ed.) *Handbuch der mechanischen Verfahrenstechnik: Partikeleigenschaften, Mikroprozesse, Makroprozesse, Zerteilen, Agglomerieren, Trennen, Mischen, Schüttgut*; Wiley-VCH: Weinheim, Germany, 2003; ISBN 3-527-30577-7.
41. Stade, O. *Streit um Gipsabbau Spitzt sich zu*; Goslar. Ztg.: Goslar, Germany, 2024. Available online: https://www.goslarsche.de/lokales/konflikt-gipsabbau-biosphaerenreservat-suedharz-624696.html (accessed on 30 December 2024).

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.