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A Refinement Method for Upgrading Energy Sector-Derived Microspheres to High-Strength Products for the Drilling Industry

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Abstract

Microspheres recovered from energy sector processes, primarily as by-products of large-scale power generation, represent a valuable aluminosilicate resource with significant application potential. Their effective upgrading is essential to maximize material recovery and support circular economy strategies aimed at reintegrating industrial by-products into high-value applications. However, the heterogeneity of microspheres in terms of grain size distribution, density, and mechanical strength poses a challenge for their efficient processing and standardization. This study presents a refinement method and process line for upgrading microspheres obtained from the energy sector into high-mechanical-strength products. The proposed approach is based on the systematic fractionation of raw material, supported by the detailed characterization of feedstock from different sources and continuous control of key technological parameters. Particular emphasis is placed on optimizing separation processes and final blending strategies to ensure consistent product quality. The developed technology enables the production of microspheres meeting stringent requirements, thereby expanding their applicability in demanding industrial sectors such as drilling engineering. At the same time, the approach contributes to increased resource efficiency by redirecting a significant portion of energy sector by-products back into the economic cycle, in line with circular economy principles.

Keywords: microspheres; energy sector by-products; circular economy; material valorization; mechanical strength; particle size distribution; density separation; process optimization; waste to resource; energy sector residues

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1. Introduction

One by-product of the coal combustion process in power plants and thermal power plants is large amounts of combustion waste such as ashes and slags. A significant part of the above-mentioned waste can be successfully subjected to recovery processes [1,2]. Knowledge of the chemical and mineral composition and physical properties of fly ash and slag allows for their multi-directional use, including in construction, road construction, mining, and also less obvious industries such as agriculture [3–5]. Fly ash includes a light fraction of aluminosilicates, called microspheres. They are formed during the combustion

of hard coal in pulverized boilers, and the mechanism of their formation is very complex and not fully explained [6]. Attempts to explain this phenomenon were made, among others, by Fenelonov et al. [7] and Danilin et al. [8].

Microspheres are defined as a very fine fraction of fly ash with low apparent density (0.3–0.5 g/cm^3) [9]. Aluminosilicate particles exhibit a spherical morphology and come in shades of gray, light gray, or white. Each microsphere comprises two distinct components: a gaseous segment occupying the interior of the grain and a solid exterior forming its shell. Within, predominantly CO_2 and N_2 reside, while trace amounts of CO, O_2, and H_2O also exist [10]. The walls of aluminosilicate grains feature an amorphous arrangement with minor crystalline inclusions, while the outer shell primarily consists of SiO_2 and Al_2O_3. Minor proportions of other oxides like Fe_2O_3, CaO, and MgO are also present, albeit in smaller quantities [11,12].

This fraction can be separated from fly ash streams using wet, dry or combined separation methods. Due to its simplicity, the wet method of obtaining microspheres is used on an industrial scale, which involves collecting a layer of material floating on the surface of settling pools (lagoons) into which ash–slag mixtures are stored [13–15].

The low value of the thermal conductivity coefficient (similar to typical insulating materials) and resistance to high temperatures make microspheres a very attractive raw material. Additional advantages are low bulk and apparent density, chemical inertness and relatively good mechanical strength. Hence, microspheres are used as: an independent thermal insulation material, a component of plasters and paints, a filler for plastics, a component for the preparation of drilling fluids, a catalyst carrier, an adsorbent, and an aggregate filler for cementitious mixtures for the protection of oil and gas wells [14,16].

Microspheres are formed spontaneously without the possibility of influencing their properties at the formation stage in pulverized fuel boilers. The physical and chemical properties of microspheres depend on the properties of combusted coal, particularly the composition of the mineral matter, and the process parameters prevailing in the boiler. It is not possible to deliberately control the properties of the microspheres formed. Therefore, obtaining microspheres with the desired characteristics is mainly based on selecting the right raw material and then subjecting it to specific refining operations. A rich source of information regarding the formation, properties, and possible directions of application can be found in review papers [17,18].

Some microspheres customers do not require special parameters of this product. It is sufficient that the microspheres offered by manufacturers have the right moisture content and are in the right grain size class. The increasingly specialized applications for microspheres are placing ever higher demands on manufacturers. Raw microspheres are therefore subjected to refining processes. The aim of these processes is to obtain appropriate operating parameters of the products, primarily in terms of density, color, mechanical strength, and grain size, including determining the oversize and undersize content.

Refining operations can be simple physical operations involving the separation of microspheres from fly ash, drying and grain classification. They can also be of a mixed nature. This means that in addition to the processes mentioned above, microsphere products can be subjected to treatments such as density separation in water or lower-density liquids or chemical treatment to impart specific properties. Modifying the properties of microspheres for the needs of specific customers is constantly required.

The drilling industry is a significant customer segment for microspheres. The microspheres provided to this segment should be characterized by high mechanical strength and low density. In an analysis of possibilities to increase the strength of microspheres, two patents reported in the Russian Federation were found. Patent description RU 2509738 "Method of obtaining coated aluminosilicate microspheres" describes a method of produc-

ing aluminosilicate microspheres from fly ash from coal-fired thermal power plants for use at high hydrostatic pressures. The technical result is an increase in mechanical strength (resistance to hydrostatic pressure), ensuring suitability for use at hydrostatic pressures of 400–500 bar as a filler for cement and drilling muds in deep oil and gas wells. Patent RU 2650987, on the other hand, presents a method for selecting microspheres of assumed strength for applications at specific hydrostatic pressures. The selection of microspheres resistant to the presumed pressure is based on hydrostatic crushing (being subjected to pressure of an appropriate force value) in an aqueous environment, followed by the separation by flotation (float/sink) of undamaged microspheres with a density less than that of water (float).

The methods outlined above for effectively increasing the strength of aluminosilicate microsphere trough coatings or subjecting them to a certain pressure and separating particles with a density lower than that of water are technically feasible. However, due to the need of damaged microspheres for energy-intensive drying operations (after coating with sealant) or drying after separation in water, the cost of refinement is very high and the price of the product unattractive for customers. On the other hand, currently known refining methods do not allow very-high-strength, low-density aluminosilicate microspheres to be produced without prior interference with the particle structure i.e., trough coating on the microsphere surface with special preparations or by partial degradation through the application of high pressure, followed by the separation of the lighter-than-water fraction.

There is therefore a need to modify the production of aluminosilicate microspheres so as to obtain high-strength microspheres without the need for additional water separations and drying. In order to avoid energy-intensive microsphere refining operations, it was assumed that the focus should be on modifying and optimizing the grain classification process. A key issue for the efficient control of the quality of the resulting commercial products in the refining process is knowledge of the properties of raw materials and the relationship between them.

This paper presents the results of implementing the research outcomes described in [19] in an industrial environment. Having previously identified the basics of the relationships between the key properties of the tested microspheres, the current work focuses on large-scale testing using an existing industrial-scale installation. The installation was modified in a manner intended to improve the functional properties of the obtained microspheres while maintaining economic feasibility, understood here as avoiding significant increases in production costs. The primary objective of these modifications was to increase the yield of microspheres with enhanced mechanical strength.

2. Materials and Methods

2.1. Methods for Evaluation of Properties of Tested Microspheres

The microspheres used in the presented large-scale laboratory tests were sourced from three different suppliers in Kazakhstan (Material 1, Material 2 and Material 3). They are extracted from lagoons located near various power plants. The suppliers gather the microspheres that accumulate on the surface of the lagoons, wash them, preliminarily dry them, pack them into big bags, and deliver them to customers. During the large-scale laboratory tests, the parameters necessary for evaluating the effectiveness of the refining processes were determined:

- Grain size distribution.
- Specific density.
- Mechanical strength (float/sink mass fraction).

All raw material and product samples collected for testing during the interlaboratory trials were dried to constant mass at 105 °C in a laboratory drying oven.

(1) Grain Size Distribution

Particle size distribution was determined using a set of laboratory sieves with square apertures of 500, 425, 315, 200, 160, 100, 90, 75, 56, and 40 µm. Sieving was performed using a Multiserw (Mulitiserw-Morek, Brzeźnica, Poland) laboratory sieve shaker. Approximately 100 cm^3 of microspheres was sieved for 10 min at a vibration amplitude of 20. The separated fractions were weighed with an accuracy of 0.01 g, and the content of each size class was expressed as the mass percentage of the initial sample.

(2) Specific density

Dried microspheres were separated into density fractions using liquids of different densities. Approximately 100 g samples were weighed with an accuracy of 0.01 g and transferred into an Imhoff sedimentation cone partially filled with water, then diluted to a total volume of 1000 cm^3. After thorough mixing, the suspension was left for phase separation into floating and sinking fractions. The process lasted approximately 2 h, with remixing every 30 min.

After separation, both fractions were quantitatively collected, transferred to stainless steel trays, and dried at 80 °C to constant mass. The obtained fractions were then weighed. Fractions with densities above 1 g/cm^3 were subjected to further analyses, whereas the floating fraction was additionally separated in liquids with densities of 0.9 and 0.8 g/cm^3. This procedure yielded fractions with densities of 0.9–1.0 g/cm^3, 0.8–0.9 g/cm^3, and below 0.8 g/cm^3. Liquids with densities lower than that of water were prepared by mixing water with ethanol or isopropanol in appropriate proportions. The density separation scheme is shown in Figure 1.

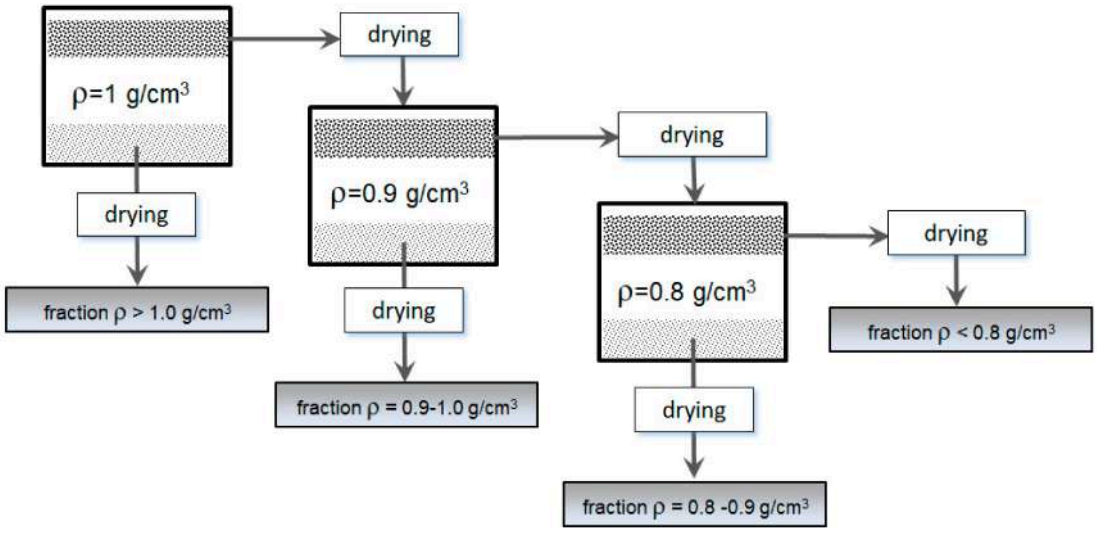

Figure 1. Procedure for density fraction separation.

(3) Mechanical Strength (Float/Sink Mass Fraction)

Mechanical strength was evaluated using a pressure resistance method commonly applied by microsphere users in the drilling industry. The test was based on the assessment of the content of sinking particles ("sinkers") after exposure to high water pressure.

The initial sinker content (*S*) was determined by dispersing 100 cm^3 of microspheres in water in an Imhoff cone filled to 1000 cm^3. After 1 h of sedimentation, the sinking fraction was collected, dried, and weighed with an accuracy of 0.01 g.

For strength testing, 100 cm^3 of microspheres was placed in a pressure vessel filled with demineralized water and subjected to a pressure of 3000 psi (20.7 MPa) for 15 min at ambient temperature. After depressurization, the sample was transferred to an Imhoff

cone for float–sink separation. Damaged particles and pre-existing sinkers settled at the bottom, whereas intact microspheres floated on the surface. The sinking fraction was dried and weighed with an accuracy of 0.01 g. Mechanical strength (*MS*) was calculated using the following formula.

$$MS = 100 \cdot \frac{m_f}{m_s} - S, \ \%$$

where

- m_f—mass of sinking particles after the mechanical strength test, g.
- m_s—mass of the microsphere sample before the mechanical strength test, g.
- S—initial content of sinking particles in the sample, %.

The acceptable mechanical strength of microspheres depends on industrial requirements. In drilling applications, microspheres with *MS* values below 30% are considered suitable, values between 30 and 40% are conditionally accepted, and materials with *MS* values above 40% are regarded as unsuitable.

2.2. Method of Upgrading Microspheres in Large-Scale Installation

The technologies for upgrading raw microsphere materials involve a combination of simple unit operations such as drying, sieving, and possibly mixing. The proper configuration of the technological line, tailored to the quality of the processed raw material, enables the achievement of the intended product parameters. Figure 2 shows a schematic of the microsphere upgrading plant, where different configurations of screens and transfer tubes were tested for the most favorable product quality parameters. Figure 2 also shows a modification of the upgrading plant, which is marked in green.

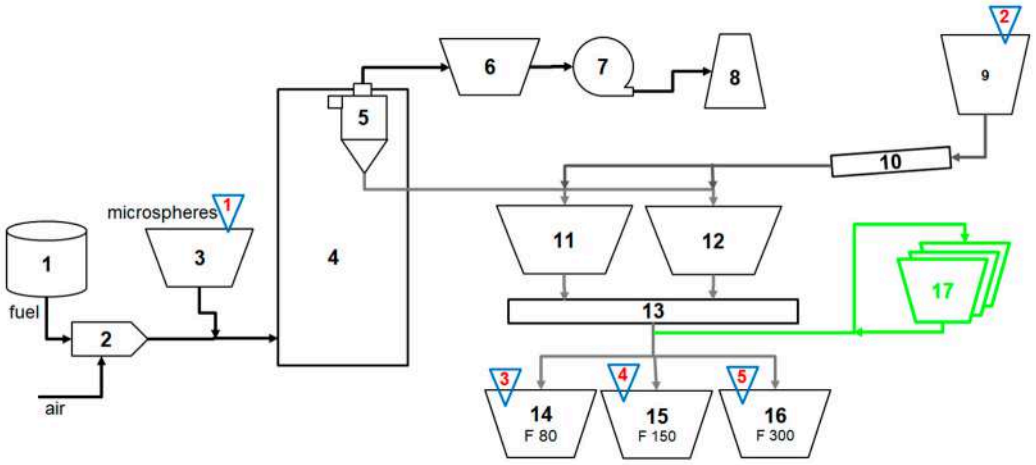

Figure 2. Schematic diagram of large-scale microsphere upgrading installation. (1)—fuel tank; (2)—combustion chamber; (3)—microsphere container; (4)—drying chamber; (5)—cyclone; (6)—bag filter; (7)—fan; (8)—chimney; (9)—hopper; (10)—pneumatic conveyor; (11, 12)—grain classification node; (13)—discharge pipes; (14, 15, 16)—collected containers; (17)—storage containers. Blue triangles mark sampling locations for laboratory tests.

The production line allows for the grain separation of wet microspheres. In the case of a wet microsphere, it is dried in a stream of hot exhaust gases obtained by burning fuel oil in the combustion chamber (2). The stream of hot exhaust gases, to which moist microspheres are dosed from the microsphere container (3), transports them to the drying chamber (4). Dried microspheres are separated from the exhaust gas stream using a cyclone (5) built in the upper part of the drying chamber. The fan (7) discharges the exhaust gases through the bag filter (6) to the chimney (8). The microspheres separated in the cyclone (5) are transported by gravity to the central element of the production line, which is the

grain classification node equipped with Allgaier screens (11 and 12). The microsphere that does not require drying is collected in the hopper (9), from where it is transported via a pneumatic conveyor (10) to the screens (11 and 12). Depending on the current demand, the screens are equipped with sieve decks with various mesh sizes. Tests carried out on a production line model in a large-scale laboratory, as well as laboratory tests, clearly showed that the separation of appropriate grain classes ensures that products with appropriate mechanical strength are obtained. Therefore, it was assumed that during large-scale tests, three products, F 80, F 150 and F 300, will be released from the microsphere raw material and collected in containers (14, 15, 16). Obtaining appropriate parameters for the separated products, i.e., mechanical strength, specific density, and oversize and undersize contents, is a key element of technology. The intended effect of controlling the yield and quality of separated grain size classes is achieved by the appropriate configuration of sieve decks with dimensions of 80, 150 and 300 μm in the screens and discharge pipes between the sieve decks (13).

The results of these studies revealed that a significant part of the raw materials received from suppliers did not allow for the achievement of the product strength expected by the microsphere customers. Among the tested raw materials, there were also those characterized by exceptionally high strength.

This led to the concept of modifying the configuration of the microsphere upgrading process line. This modification involved the addition of a unit enabling the collection/deposition of microspheres with very high mechanical strength in special storage containers (17), as shown in Figure 2—the modification is marked in green. The material (improver/additive) collected in the containers (17) can be added to products that do not meet the strength criteria, allowing for the production of a product with the desired high strength.

For the purposes of evaluating large-scale tests, a procedure for collecting microsphere samples for laboratory tests was developed. The procedure is presented in the form of a block diagram in Figure 3.

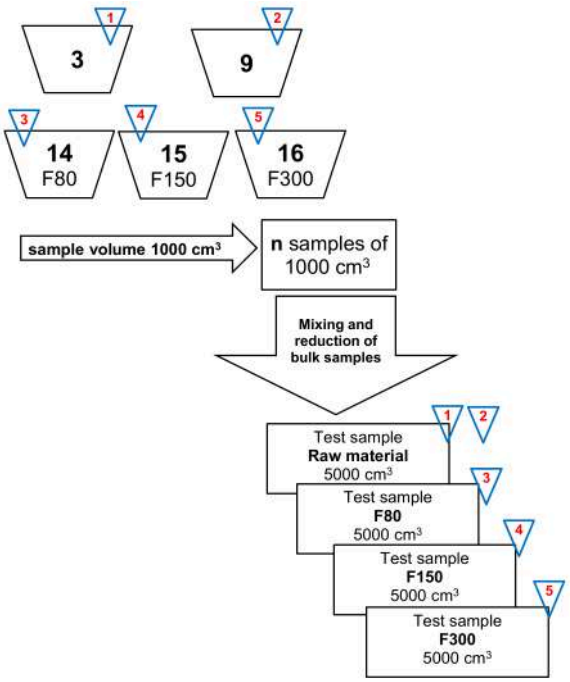

Figure 3. Collecting and preparing samples from large-scale installation for laboratory tests. (The numbering of containers and sampling locations is as shown in Figure 2).

After starting the processing of a new batch of raw material, samples were taken at regular intervals in accordance with the scheme shown in Figure 3. The individual stages of test implementation constitute the following research procedures.

1. After 60 min from the start of processing a new batch of raw material, the first series of samples in the amount of approximately 1000 cm^3 was collected in separate containers for the raw material (feed) and all products in a given configuration, i.e., for products F 80, F 150 and F 300.
2. Then, at two-hour intervals, the operation of taking subsequent series of samples (portions) was repeated until the processing of a given batch of raw material was completed.
3. Samples (portions) of raw materials and products collected in containers were thoroughly mixed (averaged) and reduced to obtain an average sample with a volume of approximately 5000 cm^3.
4. The samples prepared in the above way were placed in closed packages and described. The following were determined for each of the samples thus obtained: grain size distribution, mechanical strength and specific density. The method of performing these determinations is the same as the method of testing the properties of raw materials.

3. Results and Discussion

3.1. Relationships Between Mechanical Strength, Specific Density and Grain Size of Tested Microspheres

The raw materials tested were separated into fractions with different densities and grain classes with different grain sizes. Next, in each fraction, the sinking particles (sinkers), specific density, grain size distribution, and mechanical strength (by hydraulic crushing) were determined.

The results showed that there are relationships between the mechanical strength, specific density and grain size of microspheres. Figure 4 shows the results of the basic refinement process of raw material.

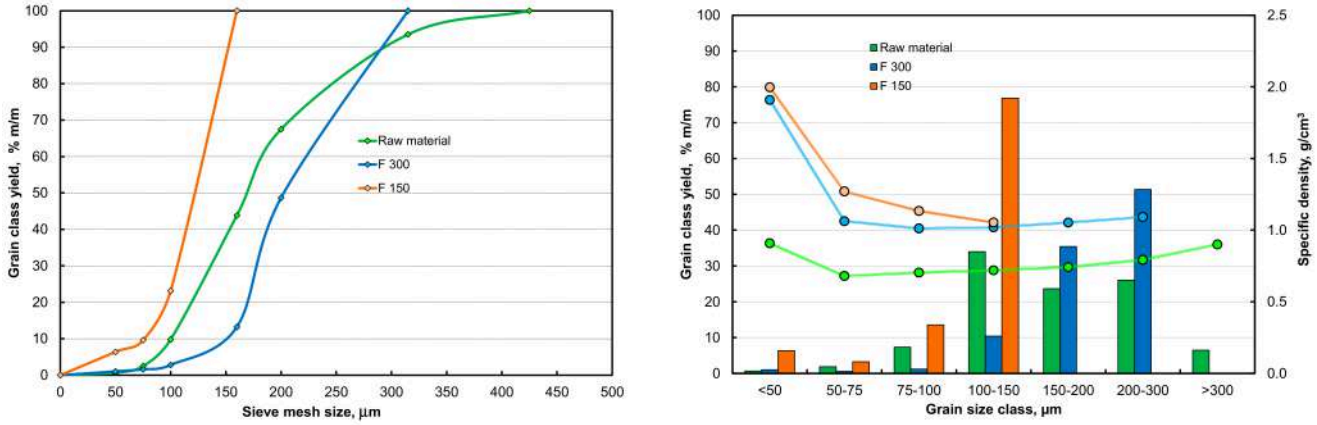

Figure 4. Microsphere raw material and two products obtained as result of basic refining processes: (**a**) grain size distribution, (**b**) yields of grain size classes and their density.

The example refining resulted in two products—F 300, containing mainly microsphere grains in the 150–300 µm range, and F 150, containing microsphere grains smaller than 150 µm. The grain distributions of the raw material and products F 300 and F 150 are shown in Figure 4a. The presented grain distributions of products F 300 and F 150 show that the grain classification process proceeds with a certain degree of efficiency, with the consequence that grains with dimensions larger (so-called oversize) or smaller (so-called

undersize) than the assumed limit values end up in the products. Figure 4b shows the yields of the individual grain size classes in the raw material and products F 300 and F 150, as well as the specific densities in the separate grain size classes. From the data presented in Figure 4b, it can be seen that the densities in the size range 75–300 µm remain at similar values, with the densities of the products being slightly higher. In contrast, a sharp increase in densities is observed for the finest grain classes, i.e., below 75 µm. As part of further investigations, the individual grain size classes were separated using the float/sink method, and the specific densities were determined for the resulting float and sink fractions. The results are shown in Figure 5.

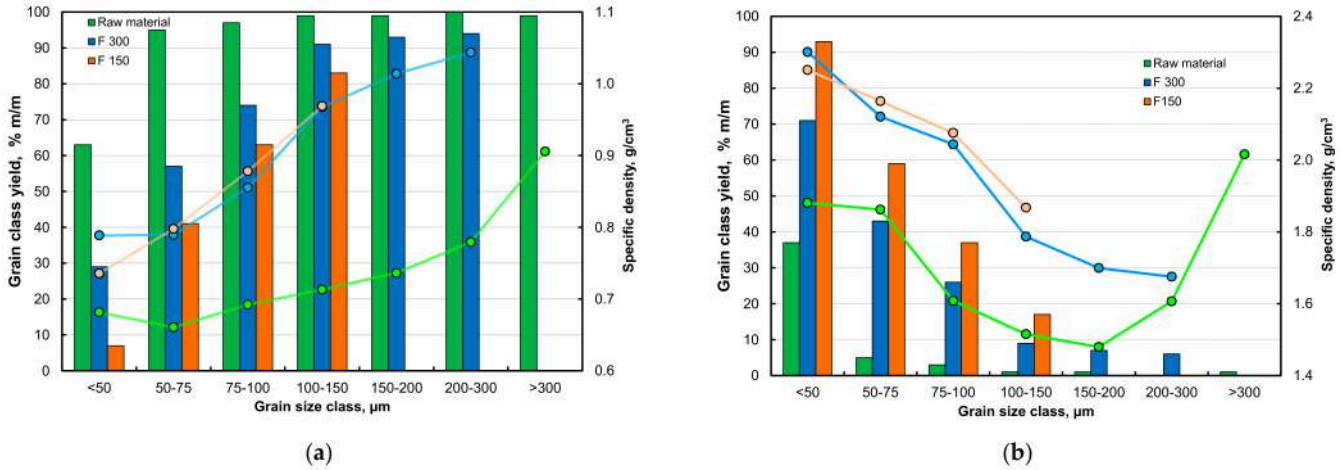

Figure 5. Yields of grain classes and their density in raw material and its refining products: (**a**) floating fraction, (**b**) sinking fraction.

The graphs show very clearly that in the case of the floating fraction (with a density lower than that of water; Figure 5a), there is a monotonic decrease in density with decreasing grain size for both the raw material and products F 300 and F 150. For the sinking fraction (Figure 5b), the relationship is reversed. For products F 300 and F 150, a clear monotonic increase in density with decreasing grain size is observed. The variation in density with grain size observed for the raw material deviates from this trend. For the raw material, we observe an initial decrease in density (for grain sizes above 200 µm), followed by an increase in density similarly to the products.

In contrast, the observed decrease in specific density progressing with decreasing microsphere size is due to the uniformity of grain morphology. The smaller the microsphere grains, the more their shape approaches that of a perfect sphere, and their walls become thinner and structurally more uniform. This effect can be observed in the microscopic images shown in Figure 6.

Paradoxically, the fine grain classes consist of a mixture of "ideal" microspheres with very low density and degradation products of various microsphere grains with very high density, which cannot be effectively separated by sieving.

In the case of large grains (above 300 µm), their walls are thick and porous (Figure 7). This means that the porous walls resulting from the crushing/destruction of large microspheres do not sink in water (they have high buoyancy/floating power). Therefore, the coarse grain classes have a higher specific density, but there are no extreme differences in density between the whole microspheres and the walls of degraded grains.

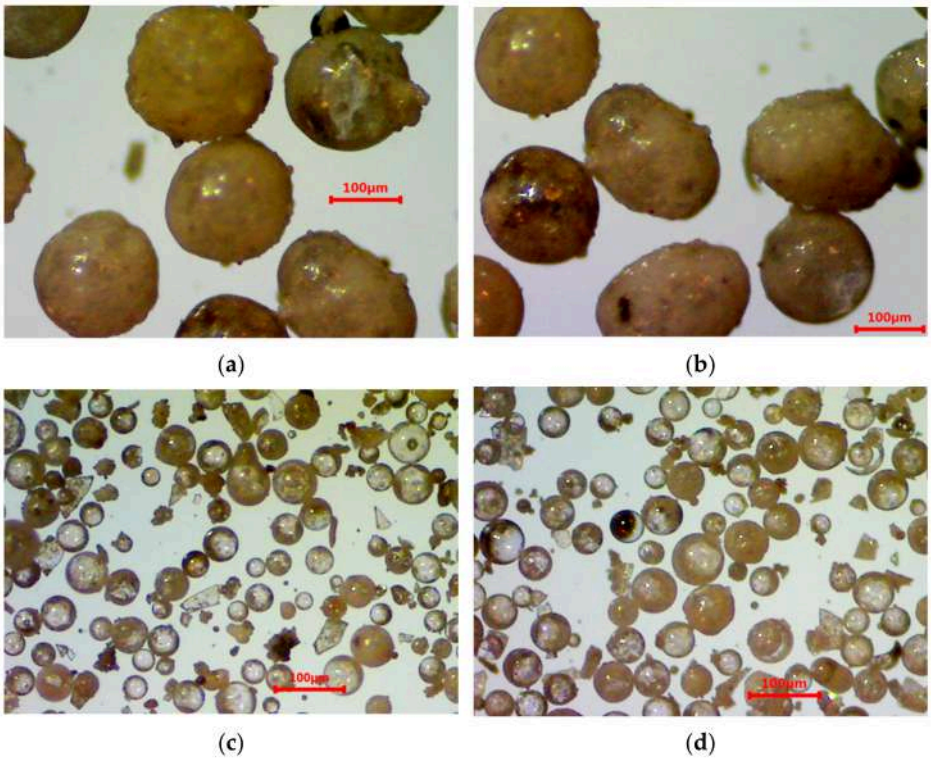

Figure 6. Transmitted light optical microscope images of the tested microspheres: (**a**,**b**)—grain size class 150–200 µm; (**c**,**d**)—grain size class < 75 µm.

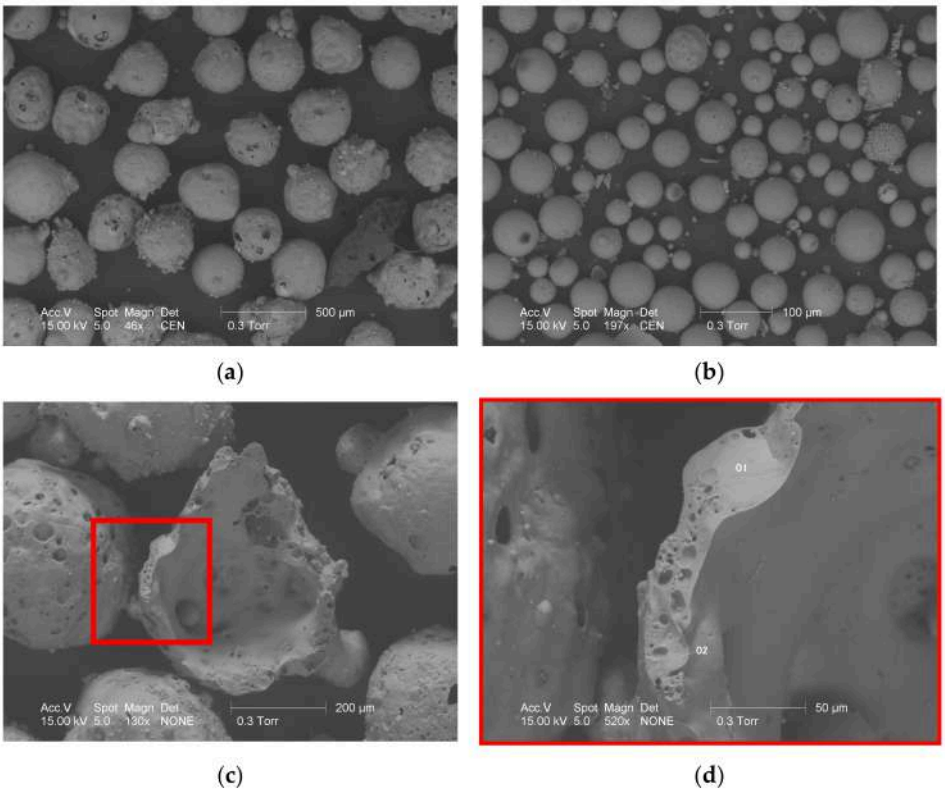

Figure 7. SEM images of tested microspheres: (**a**)—large grain habit; (**b**)—small grain habit; (**c**)—grain surface texture; (**d**)—porous structure of grain wall.

3.2. The Large-Scale Test

When analyzing the test results, attention was paid to the effectiveness of screening. The effectiveness of the separation of grain classes is assessed on the basis of the content

of grains in the assumed grain classes with dimensions lower than those desired in the assumed grain class, the so-called undersize class, and higher than desired, the so-called oversize class. For example, for the F 300 product, where we assume the desired grain size range is 150–300 µm, the undersize class consists of grains with dimensions below 150 µm, while the oversize class consists of grains with dimensions above 300 µm.

The raw Material 1 was wet and passed through the drying line and then through the grain classification line. Table 1 summarizes the results of sieve analysis for the tested microsphere raw materials and products resulting from the separation of grain classes into specific products F 80, F 150 and F 300. The following assumptions were made in this study: for the F 80 product, the grain class covers sizes in the range of 0–75 µm; for the F 150 product, the grain class covers sizes in the range of 75–150 µm; and for the F 300 product, the grain class covers sizes in the range of 150–300 µm.

Table 1. Grain size distribution of raw materials and products obtained from them.

Grain Class, µm	Share of the Grain Class, % m/m											
	Material 1				Material 2				Material 3			
	RM	F 80	F 150	F 300	RM	F 80	F 150	F 300	RM	F 80	F 150	F 300
>300	7.18	0	0	0.30	1.57	0	0	0.29	1.4	0	0	0.10
200–300	10.47	0	0	12.11	2.90	0	0	6.90	11.50	0	0	50.20
150–200	38.16	0	0.29	76.76	21.19	0	0.10	80.82	13.00	0	0.30	37.70
100–150	28.55	0	79.34	9.06	40.79	0	75.38	11.85	43.00	0.5	79.70	10.80
75–100	7.44	35.67	11.20	0.66	14.81	46.89	15.25	0.14	16.10	68.1	12.40	0.5
56–75	3.89	23.46	3.96	0.49	10.34	27.58	5.77	0	7.50	17.25	4.85	0.30
40–56	2.47	20.83	2.76	0.32	6.19	19.37	2.61	0	5.40	10.15	1.55	0.20
<40	1.84	19.64	2.45	0.30	2.20	6.16	0.89	0	2.1	4.00	1.20	0.20
OG	-	35.67	0.10	0.30	-	46.89	0.10	0.29	-	68.60	0.3	0.1
UG	-	0	9.17	10.83	-	0	9.27	11.99	-	0	7.6	12.00

RM—raw material; OG—oversize; UG—undersize. Grain classes appropriate for the products F 80, F 150 and F 300 are marked in blue.

In Table 1, the cells containing yields of the desired grain classes are marked in blue for products F 80, F 150 and F 300.

Based on the determined grain distributions for subsequent products, it can be concluded that for products F 150 and F 300, we achieve a good separation efficiency of grain classes with a low oversize content, below 0.3%, and a moderate oversize content of approximately 10% by mass. In the case of the F 80 product, the undersize content is zero, which results from the fact that it is the smallest grain class separated, while the oversize content reaches at least 36% by mass. This is a natural consequence of the difficulty in separating grains of the smallest dimensions.

In terms of technological aspects, it should be noted that the appropriate configuration of sieve decks on the screens and the distribution of material between the sieve decks ensure the high efficiency of obtaining F 150 and F 300 products.

Figures 8–10 show the grain size distribution curves of raw Materials 1–3 and the products obtained as a result of the upgrading process.

In Figure 8, showing the cumulative grain distribution curves, one can see the effectiveness of separating the raw material into the assumed products (grain classes). The product curves move sharply upwards, showing a very rapid increase in the value of the function (here the volume fraction) in the range of the function argument—here corresponding to the desired grain size for a given product.

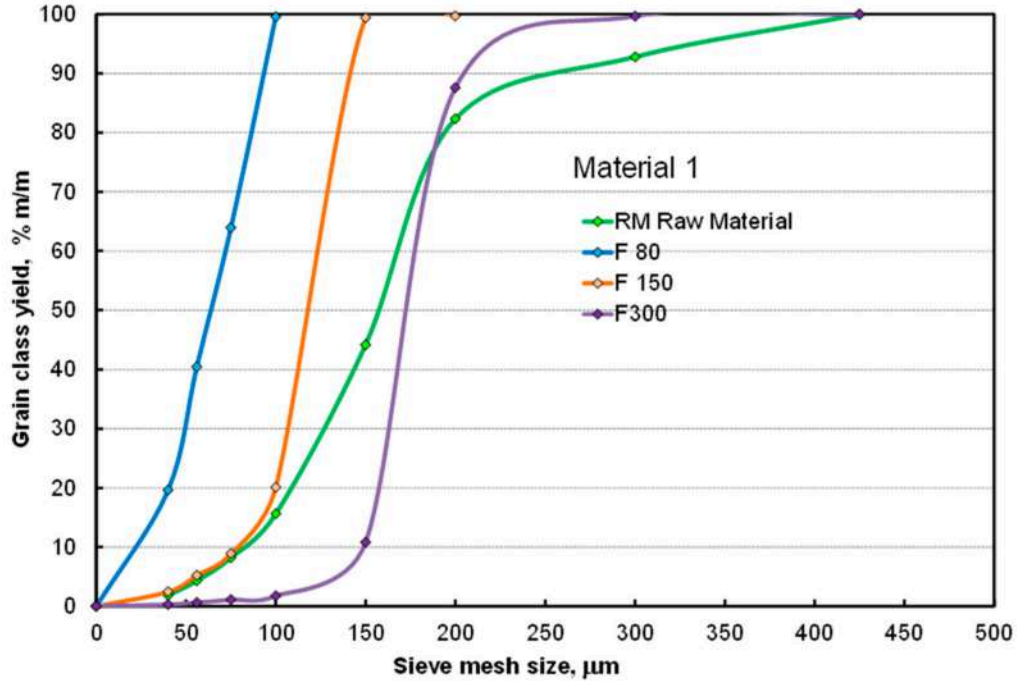

Figure 8. Grain size distribution of raw Material 1 and products obtained as result of refining processes.

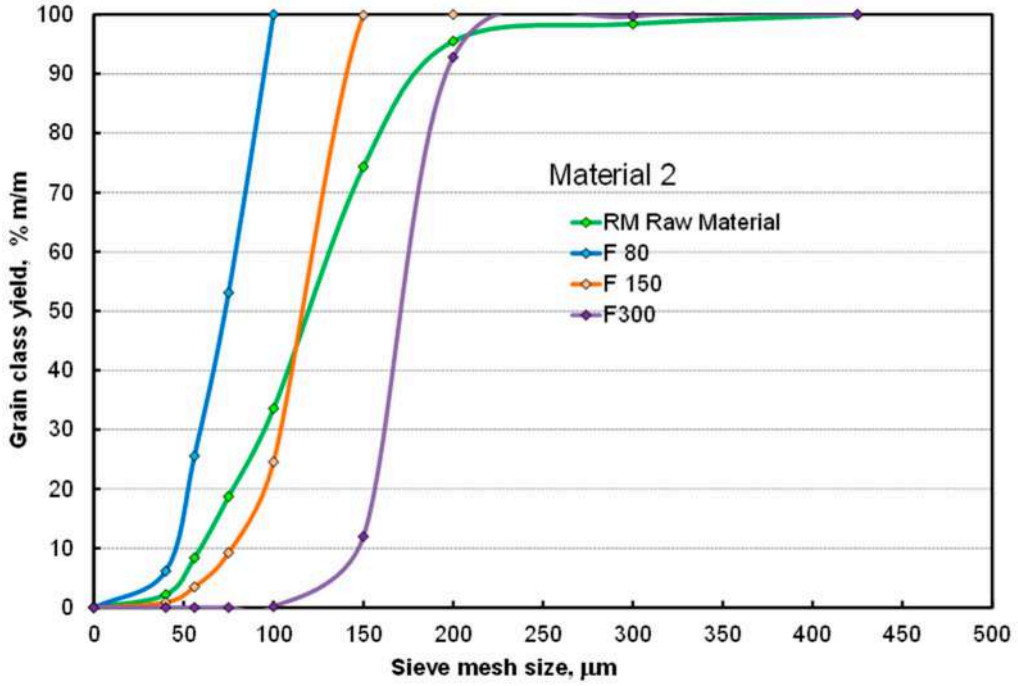

Figure 9. Grain size distribution of raw Material 2 and products obtained as result of refining processes.

Based on the data presented in Table 1 and the course of the cumulative grain distribution curves presented in Figures 8–10, it can be concluded that the grain classification process of raw Material 1 is similar to the classification of raw Material 2. Products F150 and F300 were effectively separated with a small number of excess grains (less than 0.5%) and a relatively small amount of undersize content (approx. 9–12%). The finest product (F80) contains 36% and 47% of oversize grains, which proves that the sieve beds separating the grain class with grain dimensions below 80 µm are ineffective. In the case of Material 3, it can be stated that in the case of products F150 and F300, we achieve a good separation efficiency of grain classes with a low oversize content, below 0.3%, and a moderate under-

size content of approximately 12% by mass. In the case of the F80 product, the undersize content is zero, while the oversize content reaches almost 69% by mass. It should be noted, however, that the grain class of 75–100 µm, within which the upper limit grain size of the F 80 product falls, is as much as 68% m/m. It can therefore be assumed that part of this grain class should be included in the F 80 product, and therefore the oversize content would be reduced. On the other hand, it must be taken into account that the usually high oversize fraction is a natural consequence of the separation of grains with the smallest dimensions. Similarly, one should look at the F 150 product, for which the lower grain size limit falls in the grain class of 75–100 µm. Although the mass fraction of this class is not as significant as in the case of the F 80 product, it may also increase the undersize content in the F 150 product.

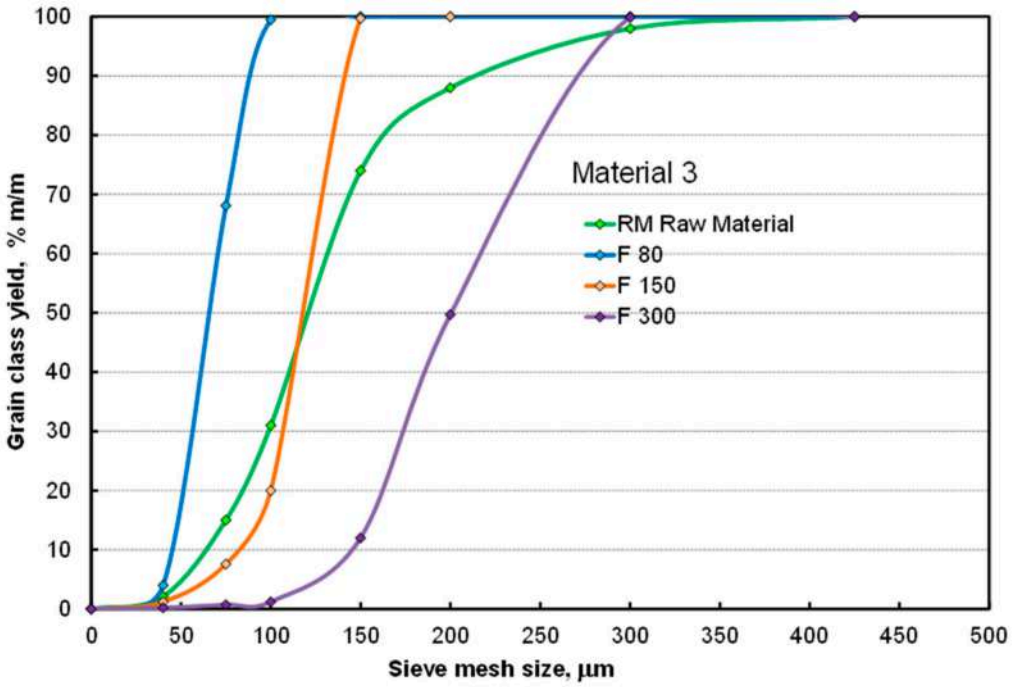

Figure 10. Grain size distribution of raw Material 3 and products obtained as result of refining processes.

Table 2 presents the results of determining the specific density and mechanical strength of raw Materials 1, 2 and 3 and products F 80, F 150 and F 300. Green indicates materials and grain classes characterized by acceptable mechanical strength.

Table 2. Specific density and mechanical strength of raw materials and grain class products.

Sample	Specific Density, g/cm^3			Mechanical Strength, % m/m		
	Material 1	Material 2	Material 3	Material 1	Material 2	Material 3
Raw material	0.9453	0.7511	0.8724	51	42	35
F 80	0.7239	0.7579	0.9159	17	33	47
F 150	0.7700	0.7621	0.9260	52	43	36
F 300	0.7848	0.8362	0.9879	59	52	24

Products with the required mechanical strength (acceptable) are marked in green.

The density values of the tested microsphere samples show great variability. This is especially visible when comparing the density of raw materials. As for the density of individual products, the tendency is that the larger the grain size, the higher the density value. In the case of mechanical strength, a difference should be noted between the quality of raw Materials 1 and 2 and raw Material 3. Only the latter is an acceptable material.

However, since the most common commercial products are grain classes of microspheres, more attention should be paid to the strength of individual grain classes. Here, similarly to the raw materials, Materials 1 and 2 show different characteristics than Material 3. For the first two cases, the best strength parameters were demonstrated for the smallest fractions, i.e., product F 80. In the case of Material 3, products F 150 and F 300 have acceptable mechanical strength, while these grain classes in the case of other microsphere materials are not high-quality products.

The relationships observed during the large-scale laboratory tests between grain size class yield and microsphere density and strength for different raw materials suggested the possibility of modifying the microsphere upgrading plant. A schematic of the modified line is shown in Figure 6.

The modification of the upgrading process involves, among others, separating the grain class of 80–150 µm from previously selected raw materials with very high mechanical strength and a minimal content of sinking particles (original sinkers). Microspheres with a density of 0.650 to 0.710 g/cm^3 and a strength index of no more than 30 (the strength index describes the mass fraction of sinking grains after the hydraulic crushing test, expressed as a percentage) are collected in separate containers marked as 17 in Figure 6. The microspheres collected in this way are used to improve the strength of refined products that do not independently meet the strength criterion (the mass fraction of sinking grains after the hydraulic crushing test exceeds 40%).

During the described series of tests, previously separated microspheres with a density of 0.68 g/cm^3 and a strength index of 29 (called an improver or additive) were used to modify the F 150 product obtained from Material 2 (Table 2). By mixing F 150 and the improver in different mass proportions, products were obtained, the properties of which are presented in Table 3. Green indicates materials characterized by acceptable mechanical strength.

Table 3. Properties of products obtained by adding improver to F 150 product from raw Material 2.

Sample	Specific Density, g/cm^3	Mechanical Strength, % m/m
Additive/Improver	0.6854	29
F 150 from Material 2	0.7621	43
90% F 150 + 10% Additive	0.7540	41
75% F 150 + 25% Additive	0.7421	38
50% F 150 + 50% Additive	0.7227	35

Products with the required mechanical strength (acceptable) are marked in green.

Overall, the results demonstrate a clear improvement over the previously applied upgrading approach. The modified process introduced the possibility of selectively recovering high-strength microsphere fractions and using them as an improver to enhance the quality of products that did not independently meet the required mechanical strength criteria. This approach not only improves the consistency and marketability of the final products but also increases the utilization of valuable raw material streams by converting fractions previously considered suboptimal into functional additives. Consequently, compared with the earlier processing method, the proposed technology provides greater operational flexibility, improved product quality control, and the ability to tailor density and strength properties to specific application requirements.

4. Conclusions

Important commercial parameters of microspheres are grain size, mechanical strength and density. With this in mind, a technological solution was proposed for separating grain classes from the microsphere raw material. The basis of the technical solution for refining

microspheres with high mechanical strength is the specific configuration of sieve decks and discharge pipes between the sieve decks. The solution used allows us to control the yield and quality of the separated grain classes, primarily in terms of grain size. The results obtained during this research indicate that the proposed technological solution for separating grain classes is characterized by screening efficiency. The effectiveness of the separation of grain classes is assessed on the basis of the content of grains in the assumed grain classes with dimensions lower than those desired in the assumed grain class, the so-called undersize class, and higher than desired, the so-called oversize class. In terms of technological aspects, it should be noted that the appropriate configuration of sieve decks on the screens and the distribution of material between the sieves ensure the high efficiency of obtaining products with larger grain sizes, namely F 150 and F 300. However, it is worth mentioning here that depending on the origin of microspheres, the actual density and mechanical strength vary.

The expansion of the laboratory and the extension of its remit enabled the systematic testing of raw materials and, above all, rapid, inter-operational control of product parameters, allowing the configuration of the process line to be adjusted to the current properties of the raw material and the assumed product quality.

As a result of testing microsphere raw materials from various suppliers, a component with very high mechanical strength was selected from these by means of advanced grain separation methods. The component is in the 80–150 μm grain class, preferably with 80% of grains of size 80–100 μm, a density of 0.650–0.710 g/cm^3 and a strength of <30% (parts sinking after the strength test). Using fraction F 150 of Material 2 as an example, the effect of adding previously separated additives, i.e., microspheres with superior functional properties, on the performance parameters of the fraction was investigated. Three additive mass fractions were applied: 10%, 25%, and 50%. It was observed that acceptable functional properties of fraction F 150 were achieved for additive contents of 25% and 50%. For these compositions, the mechanical strength values of fraction F150 were 38 and 35, respectively. Since the difference in mechanical strength between these variants was relatively small compared to the increase in additive content, the application of the lowest possible additive dosage is recommended for industrial practice. These results confirm the possibility of increasing the yield of commercial microspheres with improved mechanical strength through economically justified modifications of the existing upgrading plant.

It should also be noted that the conducted studies indicated that certain unit operations may contribute to microsphere degradation, leading to an increased content of sinking fractions. Potential causes include fluidized bed drying and pneumatic transport. Therefore, the further optimization of the beneficiation process should focus particularly on these operations.

The present work contributes to the development of an industrially applicable methodology for the beneficiation of microspheres by integrating grain size classification with quality-oriented process control based on density and mechanical strength. The proposed technological solution demonstrates that the appropriate configuration of sieve decks, material flow distribution, and the selective recovery of high-strength microsphere fractions can significantly improve the quality and commercial value of the final products. An additional contribution of this study is the identification of a high-strength improver fraction and the demonstration that its incorporation into lower-quality products enables the production of commercially acceptable microsphere products while maintaining economic feasibility. The results therefore provide a practical framework for increasing product yield, improving resource utilization, and enhancing the flexibility of microsphere upgrading plants.

At the same time, several limitations of this study should be acknowledged. The investigation was conducted using microsphere raw materials from a limited number of

suppliers, and the observed relationships between grain size, specific density, and mechanical strength may vary for materials of different origins and compositions. Furthermore, the classification efficiency for the finest fraction (F 80) remained limited due to the inherent difficulties associated with screening very fine particles. This study also indicated that certain processing operations, particularly fluidized bed drying and pneumatic transport, may contribute to microsphere degradation and increased contents of sinking particles. Consequently, the overall process performance may depend not only on classification efficiency but also on the optimization of auxiliary unit operations.

From an industrial perspective, the proposed methodology can be implemented with relatively minor modifications to existing upgrading installations. The approach relies primarily on the optimization of screen configurations, material distribution systems, and the introduction of a controlled recirculation strategy using previously separated high-strength fractions. Combined with rapid laboratory-based quality control, such a system enables the continuous adjustment of operating conditions to accommodate variations in raw material properties and target product specifications. As a result, the methodology offers a scalable and economically justified route for producing microsphere products with tailored grain size distributions, reduced density, and improved mechanical strength, thereby increasing the competitiveness and sustainability of industrial microsphere processing.

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