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DETERMINATION OF PARAMETERS FOR WET GRINDING OF PHOSPHATES IN A LABORATORY BALL MILL AND CLASSIFICATION IN A HYDROCYCLONE

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Abstract: The company Elixir Prahovo uses dry grinding of raw phosphate in its production process. However, due to increased moisture in the raw phosphate causing difficulties in the dry grinding phase, the possibility of introducing wet grinding is being considered at the industrial plant in Prahovo. The moisture content of the incoming raw phosphate varies from barge to barge, ranging between 5-10% H_2O, which is very high for the dry grinding process and create certain problems in the transportation system (the system of apron conveyors).

To assess the possibility of wet grinding of phosphates, predefined laboratory tests were conducted at the Department of Mineral Processing of the Faculty of Mining and Geology in Belgrade. This paper presents a portion of the results obtained during the aforementioned tests, which relate to the tested sample and its processing, as well as the determination of the particle size distribution of the initial sample, density, and bulk mass of phosphate ore. T In further investigations, grindability tests were conducted in a laboratory ball mill, and hydrocyclone tests were conducted on the ground phosphate in a laboratory hydrocyclone. Based on the results of these investigations, the necessary technological parameters have been determined to propose a scheme for the technological process of wet grinding and classification of phosphate. These parameters primarily relate to the material distribution in the hydrocyclone, pulp density in classification, and the particle size distribution of the ground phosphate.

Keywords: wet grinding; phosphate; classification; hydrocyclone

1 INTRODUCTION

The Chemical Products Industry (IHP Prahovo) was established in 1960, initially as a superphosphate factory and later as a producer of various granulated mineral fertilizers (Pavlica & Draškić, 1997). The founder was the Mining and Smelting Basin Bor (RTB Bor), which, aiming to address the issue of sulfuric acid neutralization, built a factory to

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convert that environmental and financial problem into a commercial product – phosphoric acid, fertilizers, and other phosphorus-based products.

In August 2012, the Elixir Group privatized IHP Prahovo, with necessary investments in the reconstruction of production and storage capacities to ensure the start of phosphoric acid production and phosphorus-based product manufacturing.

Elixir Prahovo once again takes the lead in the production of phosphoric acid and mineral fertilizers, with ambitious investment plans for the upcoming period.

In this regard, this paper also presents a portion of the research results conducted at the Department of Mineral Processing of the Faculty of Mining and Geology in Belgrade, aiming to define the scheme of wet grinding and classification of phosphates. These research efforts stemmed from the necessity to address the issue of increased moisture in the imported raw phosphate (Lazić & Nikšić, 2023).

To explore the potential of wet grinding of phosphates, a series of tests were conducted, and this paper presents a portion of those results that we consider most relevant for defining the grinding scheme and equipment selection.

Certainly, one of the crucial parameters is the particle size distribution of the feed material (Kolonja & Knežević, 2000). It is notable that the feed phosphate's coarseness is below 5 mm, and in some phosphates, up to 25% of the feed material consists of ground product. Grindability tests in the laboratory ball mill have shown that a fineness of approximately 40% passing -0.074 mm is achieved at the mill outlet in a very short time (3 minutes).

Through hydrocyclone and classification tests in the mechanical classifier, it was determined that the pulp density at the inlet to the classification process should be around 30% solids by weight, while the overflow density should be around 23-25% solids by weight (Lazić & Nikšić, 2023).

2 RESEARCH SAMPLE

A sample of Syrian phosphate weighing approximately 100 kg was provided by the expert services of the Elixir company and delivered to the Department of Mineral Processing at the Faculty of Mining and Geology in Belgrade.

In Tables 1 and 2, the particle size distribution and chemical composition of the phosphate sample used for testing are presented, along with the bulk weight with and without shaking. We can observe a moisture content of approximately 5.96%, which is very high for dry grinding (Magdalinović ,1999).

Table 1 Particle size analysis of the phosphate sample used for testing

Sample	%, H_2O	%, P_2O_5 on dry mass	Bulk weight, g/dm^3		Sieve analysis (%) mm							
			Without shaking	With shaking	1.00	0.63	0.50	0.40	0.25	0.125	0.063	<0.063
Syrian phosphate	5.96	29.43	1413.00	1692.00	16.50	6.40	4.90	5.70	21.10	27.20	9.20	9.00

Table 2 Chemical composition of the phosphate sample used for testing

% Cl	% Al_2O_3	% Fe_2O_3	% MgO	% Na_2O	% F	Cd, ppm	As, ppm	% C org
0.077	0.28	0.21	0.65	0.73	3.47	4.98	3.13	0.20

3 SAMPLE PREPARATION FOR TESTING

The processing of the initial sample of Syrian phosphate weighing approximately 100 kg was carried out in accordance with the testing program, which included homogenization, quartering, and sampling for further testing using the "checkerboard" method. One half of the sample, approximately 50 kg, was set aside as a reserve, while the other half was used for testing purposes.

On samples weighing 1 kg, the particle size distribution of the initial sample was determined through wet sieving, along with density and bulk mass determination.

In Figures 1 and 2, the "cake" before quartering and the "cake" after sampling using the "checkerboard" method are depicted.

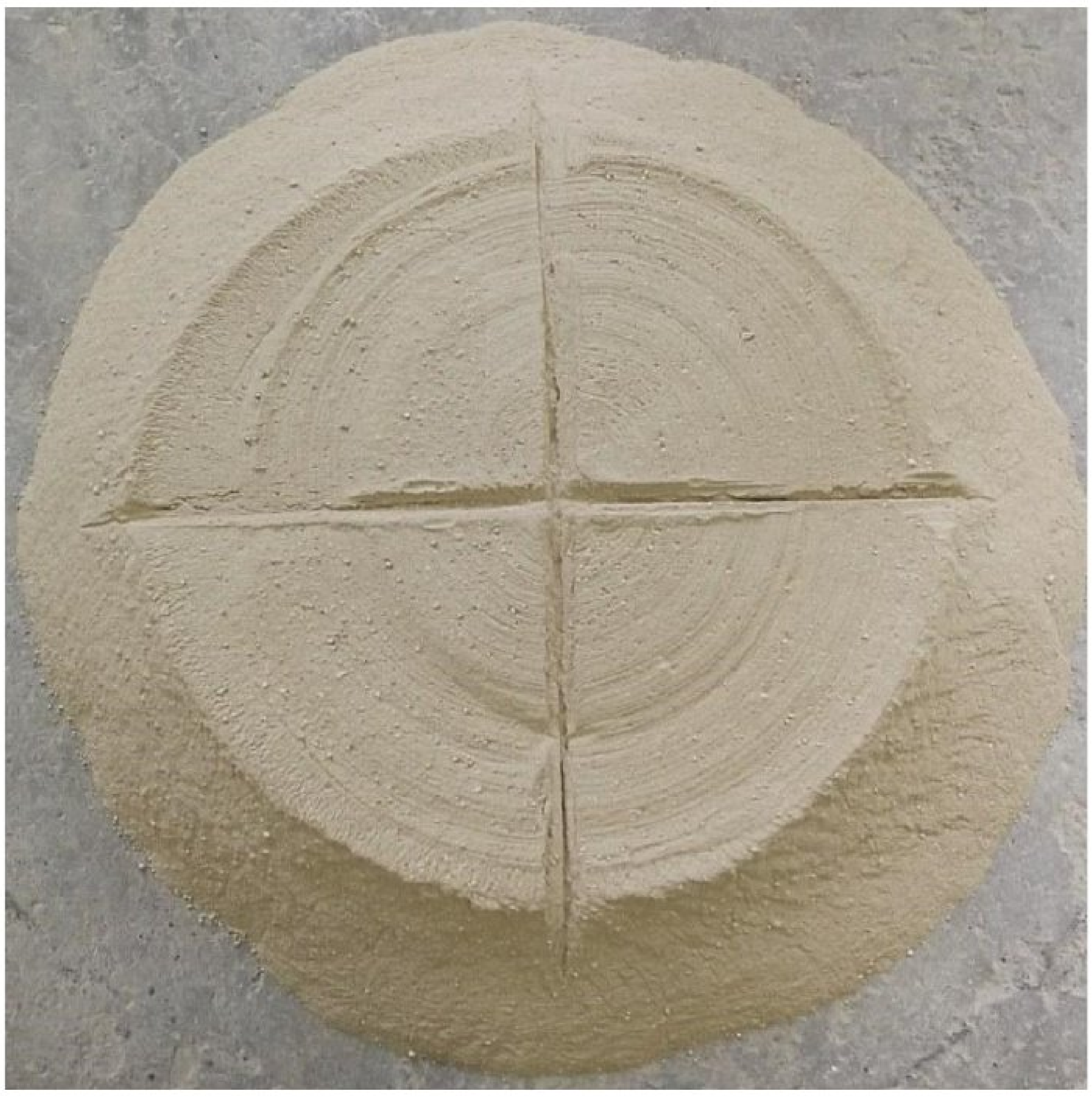

Figure 1 Cake before quartering

Figure 2 Cake after sampling using the checkerboard method

By sieving the initial sample through a series of standard sieves, the following particle size distribution was obtained:

Table 3 Particle size distribution of the initial sample before grinding

Size fraction [mm]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+3.360	3.10	3.10	100.00
-3.360 +1.651	4.40	7.50	96.90
-1.651 +0.833	5.70	13.20	92.50
- 0.833 + 0.417	10.10	23.30	86.80
-0.417 +0.297	9.70	33.00	76.70
-0.297+0.208	13.4	46.40	67.00
-0.208 +0.104	23.00	69.40	53.60
-0.104 +0.074	5.50	74.90	30.60
-0.074 +0.043	0.8	75.70	25.10
-0.043 +0.00	24.30	100.00	24.30

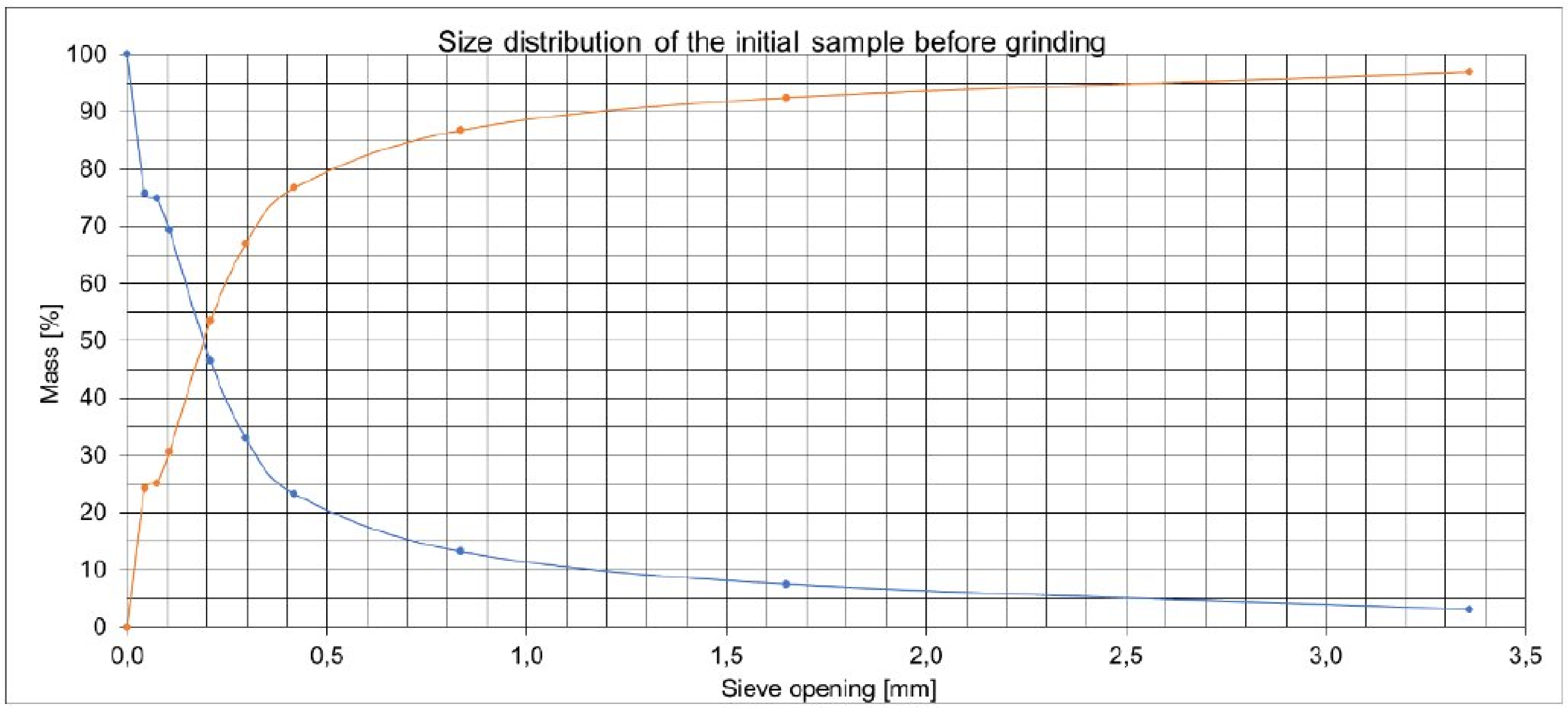

Figure 3 Particle size distribution of the Syrian phosphate sample examined in the study

Based on the particle size distribution curves, the d95 (particle size at 95% cumulative passing) of the tested phosphate is approximately 2.5 mm, with a median particle size of around 0.2 mm. The P80 (particle size at 80% cumulative passing) is 500 µm. Additionally, the percentage of the -0.074 mm class (finished product) is approximately 25%.

The density of the feed sample was determined using the pycnometer method and found to be 3.05 t/m³. The bulk mass was determined "without shaking" and amounts to 1.63 t/m³.

4 GRINDABILITY TESTS

Grindability tests were conducted using wet grinding in a Denver laboratory mill (Figure 4).

Figure 4 Denver laboratory mill (Deušić & Lazić, 2013)

The tests were conducted on samples weighing 1 kg with a ratio of solid to liquid (S:L) of 1:0.42, for durations of 5, 10, and 15 minutes. After grinding, wet sieving was performed using a 75 µm sieve, and a table and grindability curve were created, which are presented in the following text.

Table 4 Grindability of Syrian phosphate in a laboratory ball mill

Grinding time [min]	Oversieve mass [g]	Undersieve mass [g]	Size fraction [% - 74 µm]
0	749	251	25.10
5	540	460	46.00
10	421	579	57.90
15	343	657	65.70

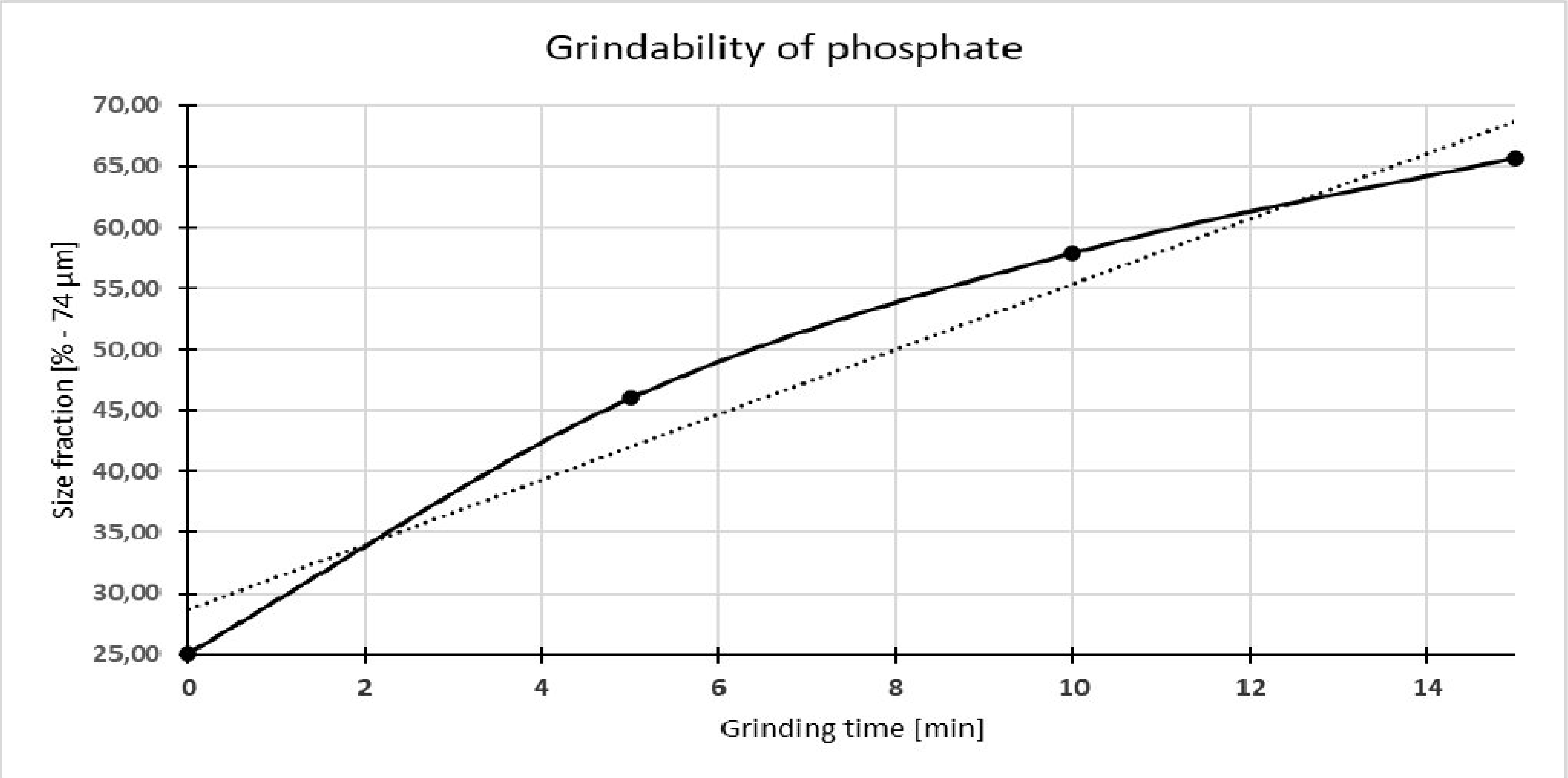

Figure 5 Grindability curve of Syrian phosphate

Based on the grindability curve, it can be observed that for a grinding fineness of approximately 40% passing -0.074 mm, the ore needs to be ground for only 3 minutes. This is understandable because the initial ore sample already contains about 25% of the ground product. The fineness of 40% passing -0.074 mm is determined based on literature data and experience with wet grinding of lead and zinc ores.

5 HYDROCYCLONE TESTS

Hydrocyclone tests on wet-ground phosphate were conducted on a laboratory setup depicted in Figure 6.

Figure 6 Laboratory setup for hydrocyclone testing

The tests were conducted by wet grinding 10 samples of 1 kg each in the laboratory ball mill to obtain the required ore mass for hydrocyclone testing. The test began by forming an inlet pulp with a density of 35% solid phase and taking a sample of the inlet pulp. Subsequently, classification was performed on a 100 mm diameter hydrocyclone with a sand nozzle size of 12 mm. During this process, samples of the hydrocyclone underflow (sand) and overflow were taken, their densities were measured, and their particle size distributions were determined.

Based on the particle size distributions of the feed, sand, and overflow, a mass balance was performed to separate the sand and overflow using the Grumbrecht method.

Table 5 Particle size distribution of the inlet pulp (35% solids), nozzle diameter d=12 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	4.00	1.82	1.82	100.00
- 0.833 + 0.417	5.00	2.27	4.09	98.18
-0.417 +0.297	10.00	4.55	8.64	95.91
-0.297+0.208	22.00	10.00	18.64	91.36
-0.208 +0.104	56.00	25.45	44.09	81.36
-0.104 +0.074	25.00	11.36	55.45	55.91
-0.074 +0.00	98.00	44.55	100.00	44.55

Table 6 Particle size distribution of the overflow (33% solids, inlet 35% solids), nozzle diameter d=12 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	0.00	0.00	0.00	100.00
- 0.833 + 0.417	2.00	0.61	0.61	100.00
-0.417 +0.297	9.00	2.75	3.36	99.39
-0.297+0.208	25.00	7.65	11.01	96.64
-0.208 +0.104	87.00	26.61	37.61	88.99
-0.104 +0.074	39.00	11.93	49.54	62.39
-0.074 +0.00	165.00	50.46	100.00	50.46

Table 7 Particle size distribution of the underflow (sand) 38% solids, (inlet 35% solids), nozzle diameter d=12 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	4.00	1.63	1.63	100.00
- 0.833 + 0.417	6.00	2.45	4.08	98.37
-0.417 +0.297	17.00	6.94	11.02	95.92
-0.297+0.208	34.00	13,88	24.90	88.98
-0.208 +0.104	65.00	26.53	51.43	75.10
-0.104 +0.074	24.00	9.80	61.22	48.57
-0.074 +0.00	95.00	38.78	100.00	38.78

Table 8 Distribution in the hydrocyclone determined by the Grumbrecht method

Size fraction [mm]	Inlet ↑	Overflow ↑	Underflow ↑				
+0.833	2	3	4	2-4	3-4	2-4x3-4	(3-4)^2
- 0.833 + 0.417	100.00	100.00	100.00	0.00	0.00	0.00	0.00
-0.417 +0.297	98.18	100.00	98.37	-0.19	1.63	0.00	3.00
-0.297+0.208	95.91	99.39	95.92	-0.01	3.47	0.00	12.00
-0.208 +0.104	91.36	96.64	88.98	2.38	7.66	18.00	59.00
-0.104 +0.074	81.36	88.99	75.10	6.26	13.89	87.00	193.00
-0.074 +0.00	55.91	62.39	48.57	7.34	13.81	101.00	191.00
+0.833	44.55	50.46	38.78	5.77	11.68	67.00	136.00
Sum						274.00	593.55

Based on the data presented in Table 8, the mass fraction of the overflow is calculated as Mpr overflow = (274 / 593.55) * 100 = 46.11%. The mass fraction of the sand is 53.89%.

After the first classification test, water was added to the cyclone pump's basket, thereby diluting the pulp to 32% solids. This was done because based on the density of the sand and overflow, it was determined that the previous classification test achieved a low classification efficiency (around 12%). At the same time, the sand discharge opening was increased from 12 to 18 mm.

Table 9 Particle size distribution of the inlet pulp (32% solids), nozzle diameter d=18 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	4.00	2.11	2.11	100.00
- 0.833 + 0.417	5.00	2.63	4.74	97.89
-0.417 +0.297	14.00	7.37	12.11	95.26
-0.297+0.208	24.00	12.63	24.74	87.89
-0.208 +0.104	52.00	27.37	52.11	75.26
-0.104 +0.074	21.00	11.05	63.16	47.89
-0.074 +0.00	70.00	36.84	100.00	36.84

Table 10 Particle size distribution of the overflow (21% solids, inlet 32% solids), nozzle diameter d=18 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	0.00	0.00	0.00	100.00
- 0.833 + 0.417	0.00	0.00	0.00	100.00
-0.417 +0.297	2.00	0.69	0.69	100.00
-0.297+0.208	3.00	1.04	1.74	99.31
-0.208 +0.104	30.00	10.42	12.15	98.26
-0.104 +0.074	29.00	10.07	22.22	87.85
-0.074 +0.00	224.00	77.78	100.00	77.78

Table 11 Particle size distribution of the underflow (sand) 48% solids, (inlet 32% solids), nozzle diameter d=18 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	4.00	1.27	1.27	100.00
- 0.833 + 0.417	8.00	2.55	3.82	98.73
-0.417 +0.297	25.00	7.96	11.78	96.18
-0.297+0.208	51.00	16.24	28.03	88.22
-0.208 +0.104	127.00	40.45	68.47	71.97
-0.104 +0.074	34.00	10.83	79.30	31.53
-0.074 +0.00	65.00	20.70	100.00	20.70

Table 12 Distribution in the hydrocyclone determined by the Grumbrecht method

Size fraction [mm]	Inlet ↑	Overflow ↑	Underflow ↑				
1	2	3	4	2-4	3-4	2-4x3-4	(3-4)^2
+0.833	100.00	100.00	100.00	0.00	0.00	0.00	0.00
- 0.833 + 0.417	97.89	100.00	98.73	-0.83	1.27	-1.00	2.00
-0.417 +0.297	95.26	100.00	96.18	-0.92	3.82	-3.00	15.00
-0.297+0.208	87.89	99.31	88.22	-0.32	11.09	-4.00	123.00
-0.208 +0.104	75.26	98.26	71.97	3.29	26.29	86.00	691.00
-0.104 +0.074	47.89	87.85	31.53	16.37	56.32	922.00	3172.00
-0.074 +0.00	36.84	77.78	20.70	16.14	57.08	921.00	3258.00
Sum						1921.00	7259.90

Based on the data presented in Table 12, the mass fraction of the overflow is calculated as Mpr = (1921 / 7259.9) * 100 = 26.47%. The mass fraction of the sand is complemented to 100%, which is 73.53%. Better classification efficiency (around 50%) was recorded, but a high overflow fineness of 77.78% passing -0.074 mm was obtained, which could pose a problem in further processing of the phosphate. In this regard, a third classification test was conducted with a pulp density at the hydrocyclone inlet of 30% solids, while simultaneously reducing the sand discharge opening to 10 mm.

Table 13 Particle size distribution of the inlet pulp (30% solids), nozzle diameter d=10 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	2.00	1.06	1.06	100.00
- 0.833 + 0.417	14.00	7.41	8.47	98.94
-0.417 +0.297	9.00	4.76	13.23	91.53
-0.297+0.208	18.00	9.52	22.75	86.77
-0.208 +0.104	49.00	25.93	48.68	77.25
-0.104 +0.074	18.00	9.52	58.20	51.32
-0.074 +0.00	79.00	41.80	100.00	41.80

Table 14 Particle size distribution of the overflow (23% solids, inlet 30% solids), nozzle diameter d=10 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	0.00	0.00	0.00	100.00
- 0.833 + 0.417	2.00	0.52	0.52	100.00
-0.417 +0.297	5.00	1.31	1.83	99.48
-0.297+0.208	14.00	3.66	5.48	98.17
-0.208 +0.104	92.00	24.02	29.50	94.52
-0.104 +0.074	44.00	11.49	40.99	70.50
-0.074 +0.00	226.00	59.01	100.00	59.01

Table 15 Particle size distribution of the underflow (sand) 46% solids, (inlet 30% solids), nozzle diameter d=10 mm

Size fraction [mm]	Mass [g]	Mass [%]	Cumulative retention [%]	Cumulative passing [%]
+0.833	4.00	1.19	1.19	100.00
- 0.833 + 0.417	9.00	2.67	3.86	98.81
-0.417 +0.297	51.00	15.13	18.99	96.14
-0.297+0.208	62.00	18.40	37.39	81.01
-0.208 +0.104	114.00	33.83	71.22	62.61
-0.104 +0.074	26.00	7.72	78.93	28.78
-0.074 +0.00	71.00	21.07	100.00	21.07

Table 16 Distribution in the hydrocyclone determined by the Grumbrecht method

Size fraction [mm]	Inlet ↑	Overflow ↑	Underflow ↑				
1	2	3	4	2-4	3-4	2-4x3-4	(3-4)^2
+0.833	100.0	100.00	100.00	0.00	0.00	0.0	0.00
- 0.833 + 0.417	98.94	100.00	98.81	0.13	1.19	0.0	1.00
-0.417 +0.297	91.53	99.48	96.14	-4.61	3.34	-15.0	11.00
-0.297+0.208	86.77	98.17	81.01	5.76	17.16	99.0	295.00
-0.208 +0.104	77.25	94.52	62.61	14.64	31.91	467.0	1018.0
-0.104 +0.074	51.32	70.50	28.78	22.54	41.71	940.0	1740.0
-0.074 +0.00	41.80	59.01	21.07	20.73	37.94	787.0	1439.0
Sum						2277.0	4504.5

Based on the data from Table 16, the mass fraction of the overflow is calculated as Mpr = (2277 / 4504.5) * 100 = 50.56%. The sand fraction is 49.44%, with a classification efficiency of 40%. In the following Table 17 and graphically in Figure 7, the cumulative overflow screening from the classification test and the expected screening (BM product) are shown.

Table 17 Particle size distribution of the hydrocyclone overflow obtained in the laboratory of FMG at different pulp densities at the hydrocyclone inlet (30% solids, 32% solids, and 35% solids) and different sand discharge openings (10 mm, 12 mm, and 18 mm)

Sieve opening, mm	Overflow 30%Č (10 mm)	Overflow 32%Č (18 mm)	Overflow 35%Č (12mm)	Sieve opening, mm	BM product
				1.00	100.00
0.833	100.00	100.00	100.00	0.63	99.80
0.417	99.48	100.00	99.39	0.50	99.30
0.297	98.17	99.31	96.64	0.40	98.70
0.208	94.52	98.26	88.99	0.25	90.00
0.104	70.50	87.85	62.39	0.13	57.50
0.074	59.01	77.78	50.46	0.063	2.44
0.000	0.00	0.00	0.00	0.00	0.00

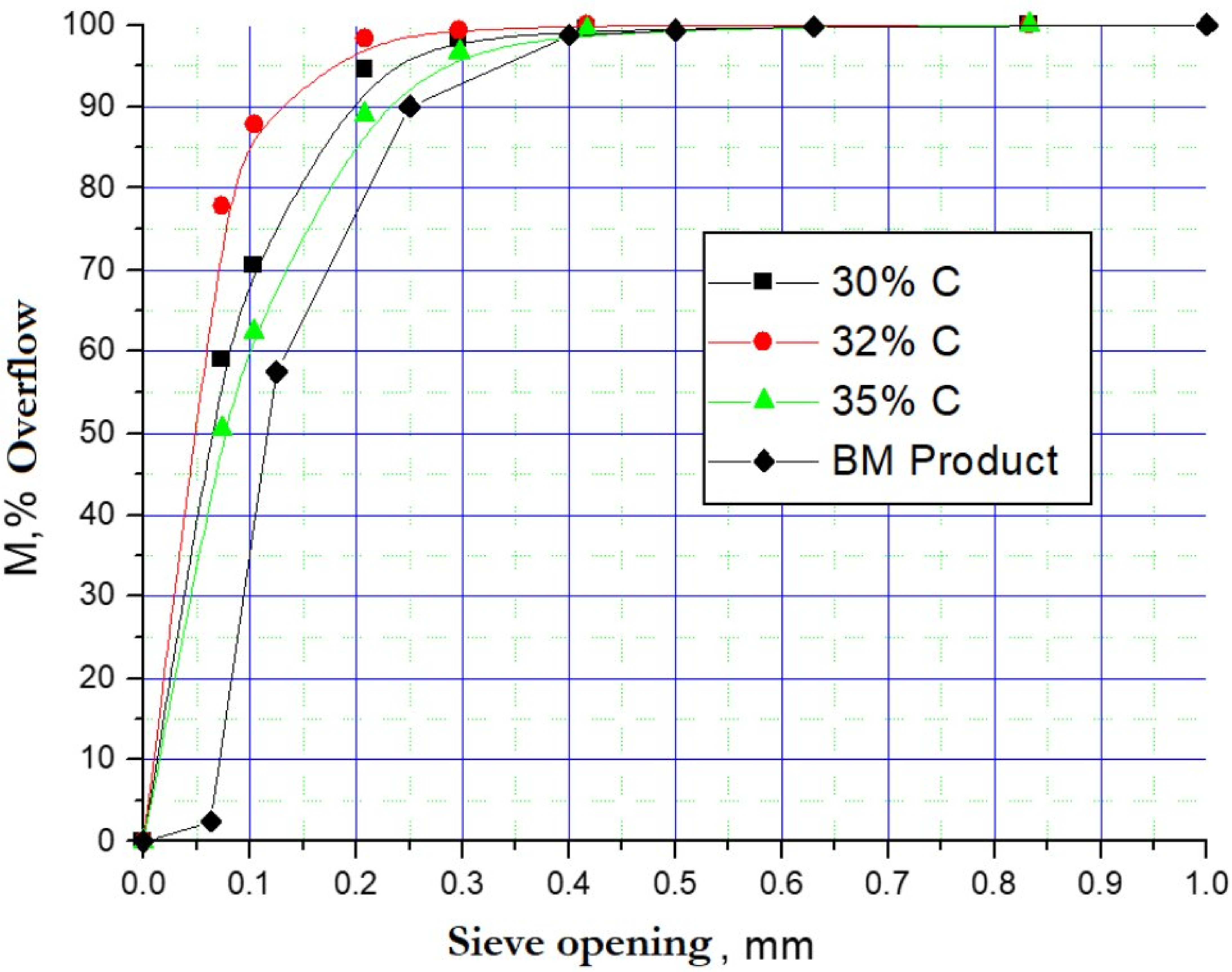

Figure 7 Cumulative overflow screening curves from the hydrocyclone and the expected curve (BM product)

Based on the overflows curves, it can be observed that all hydrocyclone overflows obtained in the FMG laboratory are finer than the expected curve (BM product). Additionally, it is evident that the hydrocyclone overflow obtained at a pulp density of 35% solids at the inlet and with a sand nozzle of 12 mm is the closest to the expected curve. For this case, the d90 of the overflow is approximately 208 µm, and the median diameter (d50) is around 74 µm. However, for this case, the classification efficiency is the lowest (12%).

For a pulp density at the hydrocyclone inlet of 32% solids and with a sand nozzle of 18 mm, the finest overflow was obtained, with a d90 of approximately 130 µm and a median diameter (d50) of around 50 µm. For a pulp density at the inlet of 30% solids and with a sand nozzle of 10 mm, the overflow with a coarseness of d90 around 200 µm and a median diameter of approximately 60 µm was obtained. In the case of the BM product curve, the d90 is around 250 µm, with a median diameter of approximately 120 µm.

6 CONCLUSION

Based on the results presented in this study, the following conclusions can be drawn:

The moisture content in the initial sample of around 6% is high for dry grinding, so considering wet grinding of phosphate is entirely justified.

The initial phosphate sample is very fine (d95= 2.5 mm) and contains approximately 25% of the -74 micrometer class (ground material), which will have a positive impact on the wet grinding process.

By wet grinding the tested phosphate sample in a laboratory ball mill for 3 minutes, the required fineness of grinding of approximately 40% passing -0.074mm is achieved, which is sufficient for the classification process in the hydrocyclone.

By classifying the ground phosphate in the hydrocyclone at different pulp densities, various finenesses of the ground product are obtained. The optimal grinding fineness of approximately 60% passing -0.074mm has been selected for further production process, which is achieved at a pulp density of 30% solids at the hydrocyclone inlet. The mass distribution of sand and overflow at this pulp density at the inlet is approximately half-half (50:50%), with a classification efficiency of around 40%.

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