

PRODUCING FEED $\text{Ca}(\text{H}_2\text{PO}_4)_2$ BASED ON ANIMAL BONE MEAL AND WET-PROCESS PHOSPHORIC ACID

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Abstract. The sustainability of agricultural and livestock production critically depends on phosphate materials, a non-renewable resource facing accelerated depletion. Animal bones can serve as an alternative source of phosphorus and calcium, with the aim of helping to meet the growing demand for feed-grade phosphates. The findings demonstrated that decomposition of calcined bone with WPA with obtaining Monocalcium phosphate that could be an efficient and low-cost strategy to improve the husbandry use of animal bone and to reduce the dependency on natural ore. Monocalcium phosphate (MCP) is a highly effective and widely used feed phosphate in the animal nutrition sector, playing a pivotal role in enhancing livestock productivity. Its widespread application highlights its importance in advancing modern animal husbandry practices. Consequently, the development of efficient and sustainable methods for producing feed-grade MCP has become a key area of research. This study explores the production of MCP through the reaction of animal bone residues with concentrated wet-process phosphoric acid (WPA) at varying P_2O_5 concentrations (34.83%, 40.46%, 46.01%, 50.55%, 55.65% and 59.41%) in a recirculation system, aiming to optimize yield and quality. The structural and compositional characteristics of the resulting MCP samples were evaluated using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The findings confirm the feasibility of producing high-quality MCP from bone meal under controlled conditions, offering a scalable, resource-efficient and environmentally sound approach to feed phosphate manufacturing.

Keywords: Wet-process phosphoric acid (WPA), bone meal, desulfurization, granulation, tricalcium phosphate, monocalcium phosphate, livestock, composition.

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1. Introduction

Sustainability agricultural and livestock production depend on phosphate rock (RP), a non-renewable resource that is being rapidly depleted. Global demand for phosphorus (P) is projected to double by 2050 and there is mounting evidence that RP reserves are increasingly at risk (Ahmed *et al.*, 2023). In recent years, phosphorus scarcity has been identified as a critical bottleneck not only for the sustainability of agricultural systems but also for livestock production (Cordell *et al.*, 2011; Chowdhury *et al.*, 2018). According to FAO statistics, global meat consumption reached 3,024 million tonnes in 2013, with an annual growth rate of 2.4% and pork and poultry comprising half of this total. Bones, accounting for around 100 million tonnes of waste, are typically incinerated or landfilled,

potentially polluting the environment. However, as bones consist of hydroxyapatite, a phosphorus-containing mineral, they can serve as a sustainable source of phosphorus for recovery (Du *et al.*, 2020). Whereas, the global cattle population grew from 1.7 billion in 2017 to 1.8 billion in 2020 to meet rising meat demand, with similar increases observed in poultry, cattle and sheep populations.

In that case animal bones can be as alternative source phosphorus and calcium with purposing cover demand for feed phosphate.

Cattle bones primarily consist of the following components: mineral substances (about 60-70% of bone mass), mainly hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), a calcium-phosphate compound that gives bones their strength; organic substances (about 30-40%), primarily collagen, a protein that provides flexibility and elasticity and water (about 10-20%), which is an important component in bone tissue. These components together provide bones with strength, rigidity and flexibility, enabling them to perform their functions in the animal's body (Maxmilla *et al.*, 2021).

Animal husbandry is a cornerstone of global agriculture, playing a vital role in ensuring food security, enhancing nutritional quality, reducing poverty and driving economic growth. To strengthen food security, it is essential to increase livestock production through the adoption of modern methods, establish value-added chains, foster cooperative relationships and provide state support to the livestock sector. Expanding the feed base, promoting household livestock breeding in collaboration with large farms and processors, meeting feed demand and leveraging advanced information technologies and scientific innovations are critical steps in this direction (Toshboev *et al.*, 2024; Fangfang *et al.*, 2017).

The growth of livestock farming, poultry farming, aquaculture and other meat production sectors is a key priority, leading to a rising demand for feed phosphates. Consequently, significant efforts are being made to maximize the utilization of raw materials, develop technologies for producing feed phosphates from animal bones and phosphate resources and advance research in this field.

Feed phosphate production can be categorized into the following groups based on the type of phosphate raw material:

- Processing of animal bone residues;
- Production of elemental phosphorus from phosphoric acid solutions obtained via thermal methods;
- Extraction of phosphoric acid from phosphate raw materials using sulfuric acid;
- Synthesis of mineral fertilizers, including calcium, ammonium and sodium phosphates (Degtyarev, 2003).

The bioavailability of phosphorus in feed phosphates depends on several factors, such as phosphorus content, the Ca:P ratio, pH levels, particle size, solubility, chemical bonding and the presence of harmful elements. The chemical structure of different phosphates also plays a critical role. Table 1 provides an overview of various types of feed phosphates.

Bone meal, a mineral feed derived from the bones of slaughtered animals through degreasing and steam refining, is a natural source of high-protein amino acids, phosphorus, carnitine, serotonin and easily digestible fats. Its rich composition enhances the nutritional value of feed, promoting growth and development in young animals, improving immunity and increasing overall productivity. Bone meal is particularly rich in minerals, containing 245 g of calcium and 118 g of phosphorus per kilogram (mineral fertilizers, including calcium, ammonium and sodium phosphates (Litsova, 2004). It is

commonly added to compound feeds, silage, concentrated feeds and crushed root crops. It is also recommended for fish and fur-bearing animals. Daily dosages vary: dairy cows receive 60-200 g, calves (up to 1 year old) 15-40 g, sheep 3-5 g and poultry (chickens, ducks, geese, turkeys) 3-10 g. Typically, bone meal constitutes 1% of the total feed weight (Havelange *et al.*, 2022).

As mentioned above, this study sheds light on the investigation of monobasic calcium phosphate production from cattle bone, with the aim of developing an environmentally safe and resource-efficient method.

2. Experimental

The research focuses on wet-process phosphoric acid (WPA) and bone meal calcined at 800°C, which contains 41.02% P₂O₅ and 50.68% CaO. The primary component of the bone meal is tricalcium phosphate (Ca₃(PO₄)₂). And the initial WPA contained 18.95% P₂O₅, 0.45% F, 0.27% CaO, 0.26% MgO, 0.41% Fe₂O₃, 0.56% Al₂O₃ and 2.96% SO₃. For its purification, strontium carbonate of grade “chemical pure” (not less than 98% SrCO₃) was used for desulfurization, while sodium carbonate (not less than 98% Na₂CO₃) was employed for defluorination.

Evaporation of purified WPA (from 34.83%, 40.46%, 46.01%, 50.55%, 55.65% and 59.41% P₂O₅) was carried out under vacuum at a pressure of 0.65 mm Hg. The chemical composition of the purified and evaporated WPA samples is given in Table 1.

Table 1. Composition of samples of purified and evaporated wet-process phosphoric acid

Concentration P ₂ O ₅ , %	Content of components, wt. %					
	CaO	MgO	Fe ₂ O ₃	Al ₂ O ₃	SO ₃	F
34,83	0,04	0,3	0,55	0,8	0,07	0,18
40,46	0,035	0,28	0,5	0,7	0,06	0,17
46.01	0.03	0.37	0.78	1.01	0.08	0.19
50.55	0.03	0.32	0.9	1.34	0.09	0.18
55.65	0.039	0.51	1.25	1.28	0.12	0.20
59.41	0.025	0.4	0.34	0.1	0.15	0.18

Table 2. Technical properties of wet-process phosphoric acid (WPA) at different temperatures and P₂O₅ concentrations

P ₂ O ₅ , %	Density, g/cm ³				Viscosity, cPz			
	60°C	70°C	80°C	90°C	60°C	70°C	80°C	90°C
34.83	1,4830	1,4765	1,4740	1,4690	3,77	3,25	3,02	2,88
40.46	1,5021	1,4910	1,4870	1,4820	4,15	4,01	3,42	3,04
46.01	1,5150	1,5080	1,5020	1,4950	5,82	4,50	3,73	3,15
50.55	1,5640	1,5500	1,5350	1,5210	8,55	6,69	5,36	4,13
55.65	1,6930	1,6770	1,6630	1,6480	23,53	17,60	14,42	11,94
59.41	1,8930	1,8850	1,8760	1,8680	80,90	55,66	40,40	29,36

Technical properties WPA can be presented in Table 2. The table demonstrates that both density and viscosity of WPA are strongly dependent on P₂O₅ concentration and temperature. As the P₂O₅ content increases from 34.83% to 59.41%, the density shows a progressive rise from 1.4830 g/cm³ to 1.8930 g/cm³ at 60°C. Similarly, viscosity exhibits a more pronounced increase, escalating from 3.77 cPz to 80.90 cPz at 60°C for the same concentration range. The data also reveal that temperature has an inverse relationship with both properties - higher temperatures result in lower density and viscosity values for any

given P_2O_5 concentration. These technical characteristics are critical for process optimization in industrial applications, particularly for the production of feed-grade monocalcium phosphate where acid handling and reaction kinetics are significantly influenced by these physical properties.

The decomposition of bone meal with WPA was conducted according to the following stoichiometric reaction, using acid dosages of 80%, 85%, 90%, 95%, 100%, 105% and 110% to ensure the formation of monocalcium phosphate (MCP):



This reaction involves the interaction of tricalcium phosphate in the bone meal with phosphoric acid, yielding monocalcium phosphate - a highly soluble and bioavailable form of phosphorus widely used in animal feed. The study aims to optimize the acid dosage and concentration to maximize the efficiency of bone meal decomposition and ensure the production of high-quality MCP.

Based on preliminary experimental data, the granulation of MCP was conducted at recirculation ratios (fine product with a particle size of less than 1 mm) of Product: Recirculation = 1:0.3, 1:0.5 and 1:0.7. These ratios were selected to optimize mixing and pelletizing conditions, ensuring a yield of the commercial product (1-4 mm) of at least 70%.

Prior to the granulation process, WPA with a concentration of 40.46 % P_2O_5 was preheated to 80°C for 15 minutes in a porcelain beaker placed in a thermostat. Subsequently, the required quantity of bone meal was gradually introduced into the preheated WPA. The reaction mixture was thoroughly mixed for 20 minutes to ensure homogeneity. Granulation was achieved through pelletizing and mixing the wet product, followed by drying the resulting MCP granules at 95-100°C for 3 hours. For each acid dosage tested, a corresponding product was prepared and utilized as recirculation material, as detailed in Table 3.

Table 3. Composition of retur monocalcium phosphate samples depending on the rate and concentration of thermal phosphoric acid

Norm WPA, %	pH of the 10% product solution	Moisture content of the product, %	Component content, mass %									
			$\text{P}_2\text{O}_{5\text{dig}}$ (soluble in 0.4% HCl)	P_2O_5 _{wait}	CaO _{tot}	CaO_{dig} (soluble in 0.4% HCl)	CaO _{wait}	MgO _{tot}	Fe_2O_3 _{tot}	Al_2O_3 _{tot}	SO_3 _{tot}	F
1	2	3	4	5	6	7	8	9	10	11	12	13
WPA concentration - 34.83% P_2O_5												
Second grade												
110	3.72	2.12	51.17	46.19	23.15	22.94	17.19	0.27	1.18	1.64	0.07	0.19
105	3.81	2.03	50.88	45.65	23.64	23.51	16.93	0.26	1.16	1.60	0.07	0.19
100	3.86	1.89	50.14	44.09	24.11	23.97	16.68	0.26	1.14	1.58	0.07	0.18
95	3.92	1.57	49.57	42.85	24.58	24.30	16.45	0.25	1.12	1.57	0.07	0.17
90	3.94	1.31	49.15	42.13	25.03	24.86	16.02	0.24	1.08	1.54	0.06	0.17
85	3.97	1.13	48.99	41.21	25.60	25.38	15.29	0.23	1.05	1.49	0.06	0.16
80	4.05	0.95	48.92	39.77	26.16	26.07	15.16	0.22	1.04	1.45	0.05	0.16
WPA concentration - 40.46% P_2O_5												
Second grade												
110	3.19	1.34	51.19	42.86	23.25	23.17	16.46	0.23	1.01	1.42	0.07	0.17
105	3.25	1.24	50.75	42.08	23.65	23.51	16.26	0.22	0.98	1.38	0.07	0.17
100	3.29	1.13	49.87	41.77	24.33	24.13	16.05	0.22	0.96	1.33	0.06	0.16
95	3.37	0.91	49.52	41.56	24.89	24.66	15.91	0.21	0.94	1.27	0.06	0.16
90	3.43	0.83	49.04	40.68	25.19	25.08	15.73	0.20	0.91	1.25	0.06	0.15
85	3.47	0.75	48.82	40.11	25.71	25.55	15.24	0.19	0.87	1.23	0.05	0.14
80	3.66	0.64	48.73	39.37	26.44	26.36	13.99	0.18	0.85	1.20	0.05	0.13

WPA concentration - 46.01% P ₂ O ₅												
Second grade												
110	3.06	2.15	51.25	41.98	23.32	23.09	15.69	0.34	0.63	0.85	0.07	0.15
105	3.08	2.01	50.85	41.55	23.57	23.49	15.03	0.33	0.63	0.84	0.07	0.15
100	3.10	1.54	49.99	40.64	24.41	24.22	14.73	0.32	0.62	0.81	0.06	0.15
95	3.12	0.99	49.57	38.97	25.04	24.86	14.38	0.31	0.62	0.80	0.06	0.14
90	3.17	0.91	49.38	38.51	25.25	25.13	14.05	0.30	0.60	0.78	0.05	0.14
85	3.21	0.86	49.22	38.02	25.93	25.68	13.85	0.28	0.58	0.75	0.05	0.13
80	3.25	0.82	49.15	37.17	26.57	26.42	13.44	0.27	0.56	0.74	0.05	0.12
WPA concentration - 50.55% P ₂ O ₅												
Second grade												
110	3.10	2.32	51.34	41.24	23.35	23.26	14.05	0.31	0.66	1.06	0.09	0.15
105	3.12	2.13	50.98	41.01	23.62	23.55	13.79	0.28	0.65	1.04	0.09	0.14
100	3.15	1.62	50.47	40.18	24.53	24.61	13.42	0.27	0.65	1.03	0.08	0.14
95	3.16	1.16	50.31	38.51	25.24	25.18	13.06	0.25	0.64	1.02	0.08	0.13
90	3.19	1.02	50.06	38.16	25.29	25.33	12.98	0.23	0.63	0.98	0.07	0.13
85	3.22	0.92	49.88	37.94	26.01	25.91	12.92	0.22	0.61	0.97	0.07	0.12
80	3.28	0.85	49.68	37.05	26.64	26.54	12.85	0.22	0.60	0.94	0.06	0.12
WPA concentration - 55.65% P ₂ O ₅												
Second grade												
110	3.11	1.96	50.88	41.82	23.38	23.33	13.14	0.41	0.87	0.98	0.10	0.15
105	3.12	1.25	50.12	41.03	23.51	23.48	12.97	0.38	0.86	0.94	0.10	0.14
100	3.15	1.16	49.75	40.62	24.77	24.61	12.85	0.36	0.84	0.89	0.09	0.14
95	3.16	1.08	48.41	39.62	25.31	25.05	12.69	0.35	0.83	0.86	0.09	0.13
90	3.18	0.93	48.11	38.02	25.45	25.21	12.24	0.33	0.78	0.84	0.08	0.13
85	3.24	0.90	47.95	37.88	26.12	26.02	12.11	0.31	0.75	0.82	0.07	0.12
80	3.29	0.76	47.82	36.76	26.20	26.11	12.03	0.28	0.71	0.77	0.06	0.11
WPA concentration - 59.41% P ₂ O ₅												
Second grade												
110	3.13	1.84	51.08	40.95	23.41	23.36	13.79	0.35	0.85	0.96	0.12	0.14
105	3.15	1.41	50.15	40.09	23.68	23.51	13.46	0.31	0.82	0.92	0.11	0.13
100	3.16	0.99	49.87	38.72	24.82	24.75	13.34	0.27	0.77	0.88	0.10	0.12
95	3.19	0.79	48.58	37.08	25.43	25.13	13.24	0.25	0.75	0.85	0.10	0.12
90	3.28	0.67	48.24	36.17	25.61	25.53	13.05	0.24	0.73	0.81	0.08	0.11
85	3.31	0.61	48.11	37.01	26.34	26.19	12.98	0.21	0.71	0.77	0.07	0.11
80	3.35	0.53	47.98	36.86	26.47	26.32	12.83	0.20	0.66	0.75	0.06	0.10

The data indicate that as the dosage of 40.46% WPA increased from 80% to 105%, the proportion of assimilable P₂O₅ in the recirculated product rose from 48.73% to 51.19%. According to GOST 23999-80, the phosphorus and calcium content of these products complies with the requirements for Grade 2 monocalcium phosphate (GOST, 1981; Khoshimov *et al.*, 2024).

Consequently, these products were utilized as recirculation material - fine product - in subsequent processing. The production of the final product, granulated MCP, was then conducted using bone meal in combination with recirculation material at varying ratios of product to recirculation: 1:0.3, 1:0.5 and 1:0.7.

A comprehensive chemical analysis of both the recirculated and final products was performed to quantify various forms of P₂O₅, CaO, MgO and F, following established methodologies (Vinnik *et al.*, 1975). The total, digestable and water-soluble P₂O₅ content in both the raw materials and the synthesized MCP was determined using a photocalorimetric method. Measurements were conducted with a UV-Spectro 1900i photocalorimeter (Shimadzu, Japan) at a wavelength of 440 nm. The experimental results exhibited an error rate of $\pm 1\%$ (Khoshimov *et al.*, 2022). The pH of a 10% solution of the final product was measured using a METTLER TOLEDO FE20/EL20 device (Germany). The static strength of the granules of the feed-grade MCP samples was determined using the MIP-1 device, following a method developed at the Research Institute of Fertilizers and Insectofungicides (NIUIF) (GOST, 2004).

The samples were subjected to X-ray diffraction (XRD) analysis using a Panalytical Empyrean powder diffractometer (Netherlands). The measurement process was

computer-controlled, utilizing $\text{CuK}\alpha$ radiation ($K\alpha_1 = 1.5406 \text{ \AA}$). The X-ray tube was operated at 30 mA current and 30 kV voltage. A scanning detector was employed, moving at a constant speed of 4 degrees per minute, with data collected in the angular range of 5° to 90° in 0.02° steps ($\omega/2\theta$ coupling). The samples were placed in a rotating chamber operating at 30 rpm. The resulting X-ray diffractograms were analyzed using the ASTM international database and the mineral X-ray data tables compiled by Mikheyev (1957) and Giller (1966), ensuring accurate phase identification and structural characterization.

To investigate the surface microstructure of the synthesized samples, a scanning electron microscope (SEM) model “EVO MA 10” (Zeiss, Germany) was employed. The prepared samples were then mounted on the microscope stage and placed in a vacuum chamber. During imaging, an accelerating voltage of 20.00 kV (EHT - Extra High Tension) was applied and the working distance (WD) was maintained at 8.5 mm. High-resolution images of individual microparticles at various magnifications were acquired using the “SmartSEM” software. Additionally, the elemental composition of the samples was analyzed using an energy-dispersive X-ray spectrometer (EDS - Oxford Instruments) equipped with Aztec Energy Advanced X-act SDD technology. The results were presented as electronic images, compositional tables and graphical spectra, providing detailed insights into the sample characteristics.

3. Results and Discussion

The composition of the granulated MCP samples is presented in Table 4.

The results presented in Table 4 demonstrate that for WPA with a P_2O_5 concentration of 40.46%, the content of digestible P_2O_5 ($\text{P}_2\text{O}_{5\text{dig.}}$) increased progressively with higher acid dosages. Specifically, at a recirculation ratio of 1:0.3, the $\text{P}_2\text{O}_{5\text{dig.}}$ content rose from 48.79% to 51.25% as the acid dosage increased from 80% to 110%. Similarly, at a recirculation ratio of 1:0.5, the $\text{P}_2\text{O}_{5\text{dig.}}$ content increased from 48.83% to 51.29%, while at a ratio of 1:0.7, it increased from 48.92% to 51.39%.

A comparable trend was observed for the water-soluble P_2O_5 ($\text{P}_2\text{O}_{5\text{wat.}}$) content. At a recirculation ratio of 1:0.3, the $\text{P}_2\text{O}_{5\text{wat.}}$ content increased from 39.42% to 42.91%. For a ratio of 1:0.5, the increase was from 39.45% to 42.95% and at a ratio of 1:0.7, it rose from 39.55% to 43.03%. These findings indicate that both digestible and water-soluble P_2O_5 contents are positively correlated with acid dosage and recirculation ratio, highlighting the influence of these parameters on the final product quality.

The 2nd Grade MCP produced under laboratory conditions, characterized by its high concentrations of digestible and water-soluble P_2O_5 , is recognized as a high-quality and effective feed additive. This product is suitable for inclusion in the nutritional diets of cattle, poultry and fish, providing a bioavailable source of phosphorus to support animal health and productivity.

The interaction between bone meal and WPA at varying P_2O_5 concentrations (34.83%, 46.01%, 50.55%, 55.65% and 59.41%) and recirculation ratios (1:0.3, 1:0.5 and 1:0.7) follows a consistent pattern. However, the absolute values of the component content in the resulting products differ. The observed discrepancy between the digestible and water-soluble forms of P_2O_5 suggests the presence of dicalcium phosphate (DCP) in the product, which may influence its overall nutritional efficacy.

The interaction between bone meal and WPA with P_2O_5 concentrations of 34.83%, 46.01%, 50.55%, 55.65% and 59.41% at recirculation ratios of 1:0.3, 1:0.5 and 1:0.7 is characterized by prolonged reaction times, ranging from 1 to 3 hours. This extended

duration is associated with significant energy consumption, which may hinder the economic feasibility of the process. In contrast, the decomposition of bone meal using WPA with a concentration of 40.46% P_2O_5 at a recirculation ratio of 1:0.7 requires only 20 minutes, representing a more efficient and technologically viable approach. Consequently, WPA with a P_2O_5 concentration of 40.46% is identified as the optimal condition for this process, balancing both efficiency and energy consumption.

The products contain substantial quantities of digestible and water-soluble calcium oxide ($CaO_{dig.}$ and $CaO_{wat.}$), a critical component for bone tissue formation in living organisms. Approximately 99% of an animal's calcium reserves are stored within its skeletal system (Khoshimov *et al.*, 2024). When calcium and phosphorus are provided in adequate amounts through feed, maintaining an optimal ratio (1.5-2:1) and ensuring proper absorption, their concentrations in bone tissue remain stable and exhibit minimal variation (Turdialieva *et al.*, 2025).

Under the experimental conditions of acid dosages (80-110%) and recirculation ratios (1:0.3-0.7) using WPA with a concentration of 40.46% P_2O_5 , the resulting feed products exhibited the following compositional characteristics: total CaO ($CaO_{tot.}$): 23.28-26.55%, $CaO_{dig.}$: 23.07-26.39%, $CaO_{wat.}$: 15.01-16.55%. In addition, all products contained magnesium oxide (MgO) within the range of 0.18-0.25%. Magnesium is one of the four most essential minerals in biological systems, alongside calcium, sodium and potassium. Intracellularly, magnesium is the second most abundant mineral after potassium, underscoring its critical role in physiological processes (Fiorentini *et al.*, 2021; Hasanguliyeva *et al.*, 2024).

The products obtained at H_3PO_4 dosages of 80-110% meet the phosphorus content requirements of GOST 23999-80 for Grade 2 MCP. Specifically, at recirculation ratios of 1:0.3-0.7 and a phosphoric acid concentration of 40.46% P_2O_5 , the resulting products contain $P_2O_{5dig.}$ in the range of 48.79-51.39%, $P_2O_{5wat.}$ from 39.42% to 43.03%, $CaO_{dig.}$ between 23.28% and 26.55%, $CaO_{wat.}$ from 23.07% to 26.39% and magnesium oxide (MgO) in the range of 0.18-0.25%. Most importantly, all products have minimal fluorine (F) content-less than 0.18%, which is well below the allowable limit of 0.2%, ensuring their safety and suitability as feed additives.

Table 4. Composition of granulated feed-grade MCP samples depending on the dosage of WPA and mass ratio of product: recirculation (WPA concentration: 40.46% P_2O_5)

Norm WPA, %	pH of the 10% product solution	Moisture content of the product, %	Component content, mass %									
			$P_2O_{5dig.}$ (soluble in 0.4% HCl)	$P_2O_{5wat.}$	$CaO_{tot.}$	$CaO_{dig.}$ (soluble in 0.4% HCl)	$CaO_{wat.}$	$MgO_{tot.}$	$Fe_2O_{3tot.}$	$Al_2O_{3tot.}$	$SO_{3tot.}$	F
1	2	3	4	5	6	7	8	9	10	11	12	13
At a recirculation ratio of 1:0.3												
Second grade												
110	3.21	2.06	51.25	42.91	23.28	23.20	16.48	0.25	1.16	1.61	0.07	0.18
105	3.30	2.02	50.81	42.13	23.68	23.54	16.28	0.24	1.15	1.60	0.06	0.18
100	3.33	1.93	49.93	41.82	24.36	24.16	16.07	0.23	1.13	1.57	0.06	0.17
95	3.35	1.71	49.58	41.61	24.92	24.69	15.93	0.22	1.11	1.54	0.06	0.15
90	3.41	1.49	49.10	40.73	25.22	25.11	15.75	0.20	1.05	1.53	0.05	0.14
85	3.56	1.35	48.88	40.16	25.74	25.58	15.26	0.19	1.02	1.49	0.05	0.13
80	3.60	1.12	48.79	39.42	26.47	26.39	15.01	0.18	1.01	1.40	0.05	0.12
At a recirculation ratio of 1:0.5												

Second grade												
110	3.25	2.75	51.29	42.95	23.30	23.07	16.52	0.24	1.07	1.50	0.07	0.18
105	3.34	2.63	50.85	42.17	23.70	23.46	16.31	0.23	1.03	1.46	0.07	0.16
100	3.37	2.59	49.97	41.86	24.38	24.14	16.09	0.23	1.01	1.40	0.06	0.16
95	3.51	2.14	49.62	41.65	24.94	24.69	15.95	0.22	0.99	1.34	0.06	0.15
90	3.56	2.01	49.14	40.76	25.24	24.99	15.77	0.21	0.96	1.32	0.06	0.15
85	3.58	1.98	48.92	40.19	25.76	25.51	15.28	0.20	0.92	1.30	0.05	0.14
80	4.02	1.62	48.83	39.45	26.50	26.23	15.03	0.19	0.90	1.27	0.05	0.14
At a recirculation ratio of 1:0.7												
Second grade												
110	3.31	2.98	51.39	43.03	23.35	23.13	16.55	0.23	1.03	1.44	0.07	0.17
105	3.39	2.92	50.95	42.25	23.75	23.53	16.35	0.22	0.99	1.40	0.07	0.17
100	3.42	2.80	50.07	41.94	24.43	24.20	16.14	0.22	0.97	1.35	0.06	0.16
95	3.45	2.37	49.72	41.72	24.99	24.76	15.99	0.21	0.95	1.29	0.06	0.16
90	3.49	2.11	49.24	40.87	25.29	25.06	15.83	0.20	0.92	1.27	0.06	0.15
85	4.06	2.03	49.02	40.29	25.82	25.58	15.32	0.19	0.88	1.25	0.05	0.14
80	4.11	1.77	48.92	39.55	26.55	26.30	15.07	0.18	0.86	1.22	0.05	0.13

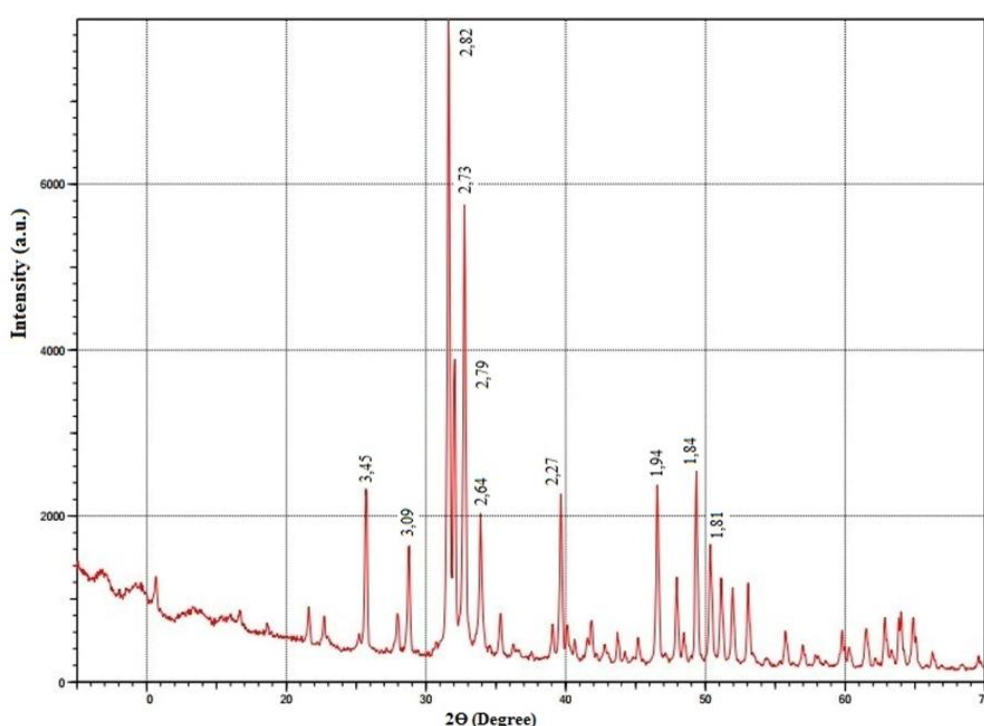


Figure 1. XRD pattern of animal bone meal

Further research should include XRD analysis of both the animal bone residues and the synthesized MCP sample derived from bone meal. This investigation will provide critical insights into the structural and phase composition of the materials, enabling a deeper understanding of the transformation process and the quality of the final product. The resulting XRDs are shown in Figures 1 and 2.

The XRD analysis of the bone meal sample heat-treated at 800 °C revealed the presence of multiple calcium phosphate phases, including β -tricalcium phosphate ($\beta\text{-Ca}_3(\text{PO}_4)_2$), fluorapatite ($\text{Ca}_5\text{F}(\text{PO}_4)_3$ and $\text{Ca}_{10}(\text{PO}_4)_6\text{F}_2$), monetite ($\text{Ca}_3(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$) and hydroxyapatite ($\text{Ca}_5(\text{PO}_4)_3(\text{OH})$). The predominant peaks at d-spacings of 3.45 Å, 2.82 Å and 2.79 Å correspond to β -tricalcium phosphate and fluorapatite, indicating their stability at high temperatures. The presence of monetite suggests partial hydration, while hydroxyapatite peaks at 2.27 Å and 1.81 Å confirm its crystalline structure (Table 5).

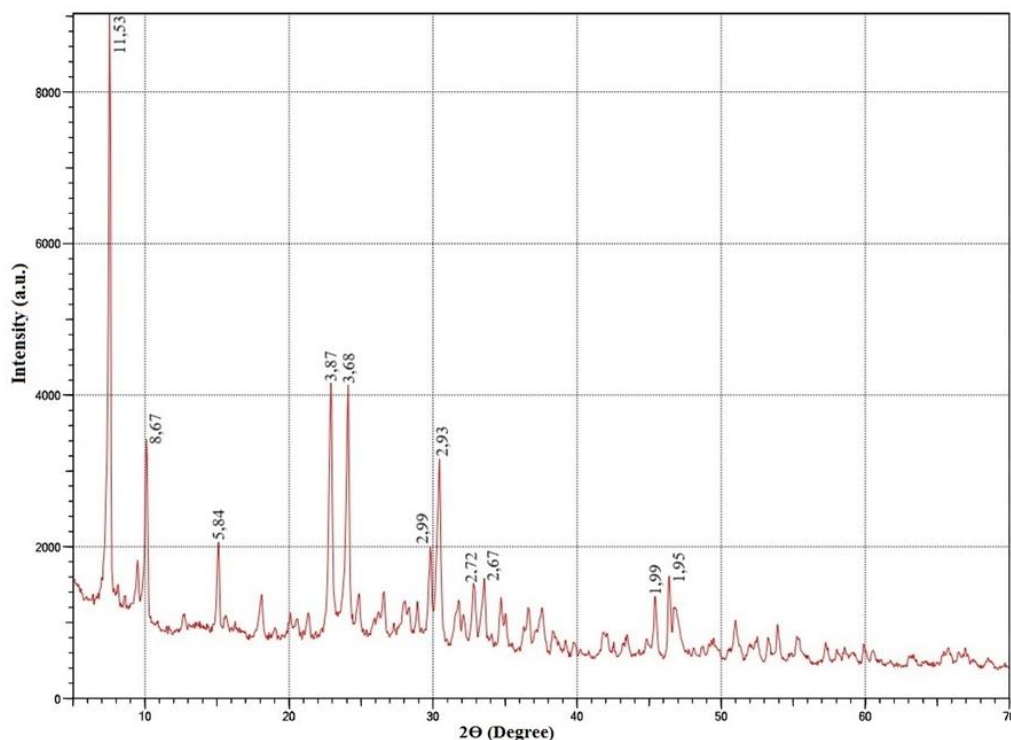


Figure 2. XRD pattern of granulated MCP sample (WPA concentration - 40.46 % P₂O₅, norm WPA - 105 %, mass ratio Product: Recirculation - 1:0.3)

Table 5. Results of XRD analysis of the initial bone raw material

No	dÅ	Corresponding substances
1	3,45	β -Ca ₃ (PO ₄) ₂
2	3,09	Ca ₅ F(PO ₄) ₃
3	2,82	Ca ₃ (PO ₄) ₂ · H ₂ O
4	2,79	Ca ₁₀ (PO ₄) ₆ F ₂
5	2,73	Ca ₅ F(PO ₄) ₃
6	2,64	Ca ₃ (PO ₄) ₂ · H ₂ O
7	2,27	Ca ₅ (PO ₄) ₃ (OH)
8	1,94	Ca ₅ (PO ₄) ₃ (OH)
9	1,84	Ca ₃ (PO ₄) ₂ · H ₂ O
10	1,81	Ca ₅ (PO ₄) ₃ (OH)

The XRD analysis of the sample revealed the presence of several calcium phosphate phases, including hydrated and anhydrous forms. The primary phases identified were Ca(H₂PO₄)₂·H₂O (monocalcium phosphate monohydrate), Ca₃(PO₄)₂ (tricalcium phosphate) and CaHPO₄ (monetite). The most prominent peaks were observed at d-spacings of 11.53 Å, 8.67 Å and 5.84 Å, corresponding to Ca(H₂PO₄)₂·H₂O and Ca₃(PO₄)₂. Additionally, peaks at 3.87 Å, 3.68 Å and 2.67 Å further confirmed the presence of hydrated monocalcium phosphate. The detection of monetite (CaHPO₄) at 2.99 Å and its hydrated form (CaHPO₄·2H₂O) at 2.93 Å indicates the coexistence of both anhydrous and hydrated calcium phosphate phases. The experimental results indicate that the predominant component of the synthesized MCP sample is MCP monohydrate, confirming that the intended product has been successfully obtained. This

outcome validates the effectiveness of the experimental methodology in achieving its objectives (Table 6).

Table 6. Results of XRD pattern of granulated MCP sample (WPA concentration - 40.46% P_2O_5 , norm WPA - 105%, mass ratio product: return - 1:0.3)

No	dA	Corresponding substances
1	11.53	$\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$
2	8.67	$\text{Ca}_3(\text{PO}_4)_2$
3	5.84	$\text{Ca}(\text{H}_2\text{PO}_4)_2$
4	3.87	$\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$
5	3.68	$\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$
6	2.99	CaHPO_4 (monetite)
7	2.93	$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$
8	2.72	$\text{Ca}_3(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$
9	2.67	$\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$
10	1.99	$\text{Ca}_3(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$

Physicochemical studies were conducted on the synthesized monocalcium phosphate products to evaluate their properties. Representative MCP samples were selected for scanning electron microscopy (SEM) analysis, as illustrated in Figure 3.

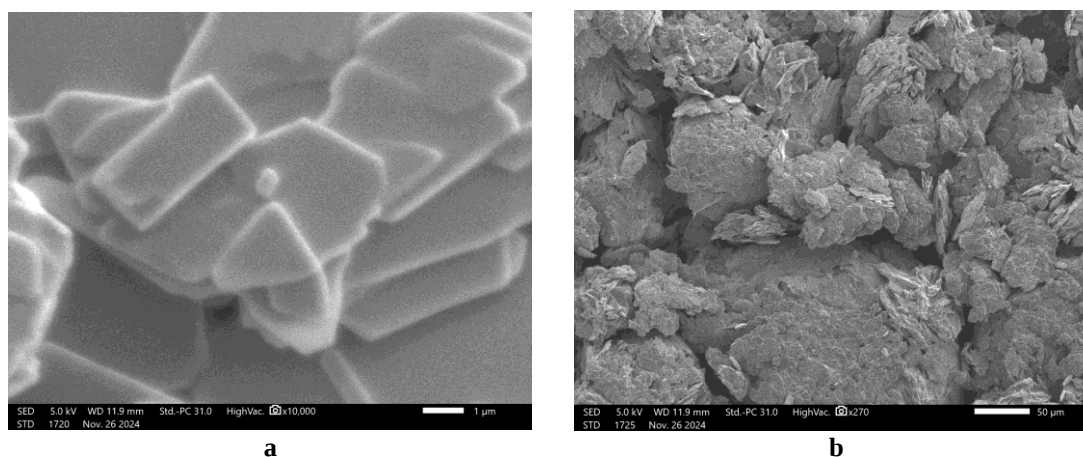


Figure 3. SEM analysis micro particles 1 μm (a) and 50 μm (b) of obtained granulated MCP sample (WPA concentration - 40.46% P_2O_5 , norm WPA - 105%, mass ratio Product: Recirculation - 1:0.3)

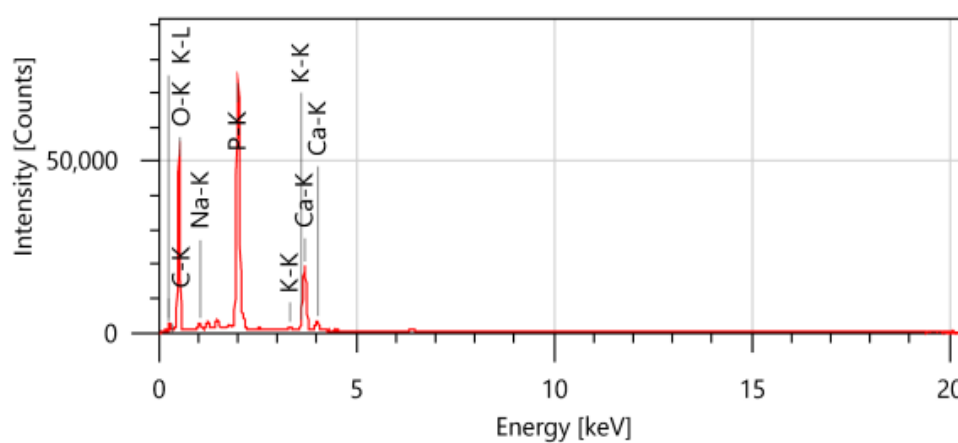


Figure 4. Results of EDS analysis of granulated MCP sample (WPA concentration - 40.46% P_2O_5 , norm WPA - 105%, mass ratio product: recirculation - 1:0.3)

The SEM images of the sample in Figure 3, acquired at magnifications of $10,000\times$ (1 μm scale - a) and $270\times$ (50 μm scale - b), reveal the microstructure and morphology of the material. Elemental analysis via energy-dispersive X-ray spectroscopy (EDS) confirmed the presence of carbon (C), oxygen (O), phosphorus (P) and calcium (Ca) as the primary constituents, with trace amounts of sodium (Na) and potassium (K). The atomic percentages of P (14.87%) and Ca (4.86%) align with the expected stoichiometry of monocalcium phosphate (MCP, $\text{Ca}(\text{H}_2\text{PO}_4)_2$), while the high oxygen content (69.25%) supports the presence of hydrated phases (Table 5). The observed microstructure and elemental composition are consistent with the formation of MCP and its hydrated forms, as further corroborated by XRD analysis. These findings highlight the material's chemical purity and structural characteristics, which are critical for its potential applications in food phosphates or biomaterials.

4. Conclusion

Thus, there is been created the feasibility of producing feed-grade MCP based on animal bone residues calcined at 800 °C and purified and evaporated wet process phosphoric acid containing various concentration ranged from 34.83 to 59.41% P_2O_5 in the presence a recirculation ratio of 1:0.3, 1:0.5 and 1:0.7. In a case, the monocalcium phosphate synthesized using phosphoric acid dosages of 80-110% fully complies with the specifications for a second grade product, confirming its suitability for use as a high-quality feed additive. Whereas, the second grade was used as a retur monocalcium phosphate product applied in a recirculation ratio. These findings indicate that both digestible and water-soluble P_2O_5 contents are positively correlated with acid dosage and recirculation ratio, highlighting the influence of these parameters on the final product quality. From a point of the technologically viable approach and economic feasibility view, optimal condition is the decomposition of bone meal using WPA with a concentration of 40.46% P_2O_5 at a recirculation ratio of 1:0.7 requires only 20 minutes. The products obtained at H_3PO_4 dosages of 80-110% meet the phosphorus content requirements of GOST 23999-80 for grade 2 MCP. Specifically, at recirculation ratios of 1:0.3-0.7 and a phosphoric acid concentration of 40.46% P_2O_5 , the resulting products contain $\text{P}_{2\text{O}_{5\text{dig}}}$ in the range of 48.79-51.39%, $\text{P}_{2\text{O}_{5\text{wat}}}$ from 39.42% to 43.03%,

SEM and XRD analyses confirm the structural and chemical integrity of the synthesized MCP, revealing a homogeneous microstructure and a phase composition primarily consisting of monocalcium phosphate and its hydrated forms. Elemental analysis demonstrates the high purity of the product, with phosphorus and calcium as the dominant elements and only trace amounts of impurities that do not affect product quality. These results underscore the effectiveness of the proposed method for producing MCP that meets industrial standards, offering a sustainable and efficient approach to valorizing bone meal residues. This study contributes to the development of advanced feed additives with potential benefits for animal nutrition and agricultural productivity.

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