RESEARCH OF PROPERTIES AND CHEMISM OF DOUBLE SUPERPHOSPHATE

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Abstract
Over the past decade, the chemical industry has undergone dramatic changes: new technologies are being introduced in practice and development has begun. These changes have led to a higher level of technological processes and devices in production. Technologies for the production of double superphosphate began to develop rapidly. Research work is aimed at improving the quality of the product, that is, its physical and chemical properties.

Keywords: superphosphate, concentration, double, crystal, suspension, granular, neutral, particle, extract.

Superphosphate is a mixture of $Ca(H_2PO_4)_2 \cdot H_2O$ and $CaSO_4$. It is the most common mineral phosphorus fertilizer. Phosphorus in superphosphate is present mainly in the form of monocalcium phosphate and free phosphoric acid. The fertilizer contains gypsum and other impurities (iron and aluminum phosphates, silica, fluorine compounds, etc.). Superphosphate is obtained from natural phosphates or apatite concentrate by treating them with sulfuric acid, according to the reaction:

$$Ca_3(PO_4)_2 + 2H_2SO_4 = Ca(H_2PO_4)_2 + 2CaSO_4.$$
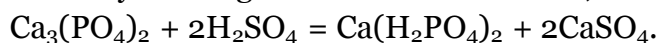
To obtain double superphosphate, apatite or phosphorite is treated with phosphoric acid.

Simple superphosphate is a gray powder, almost non-caking, medium sieving; fertilizer contains 14-19.5% of P_2O_5 assimilated by plants. It is obtained by the action of H_2SO_4 on natural phosphates (apatites, phosphorites). Granular superphosphate is obtained from simple (powdery) superphosphate by moistening and rolling it into granules (their diameter is mainly 2-4 mm) in rotating drums. It has increased disperse ability. Enriched superphosphate is obtained by decomposition of phosphate raw materials with a mixture of H_2SO_4 and H_3PO_4; in composition, it is a product intermediate between simple and double superphosphates and contains 24-32% of assimilable P_2O_5. Double superphosphate

is produced by the action of H_3PO_4 on natural phosphates. It contains 45-48% of P_2O_5 assimilable by plants, contains very little gypsum, and is produced mainly in granular form. In addition to 14-19.5% P_2O_5, ammoniated superphosphate contains at least 1.6% ammonia; manganized superphosphate contains 1.5-2.5% manganese; boric superphosphate contains 0.1-0.3% boron; and molybdenum superphosphate contains 0.1% molybdenum.

Superphosphate is used on all soils as the main pre-sowing, sowing (preferably granular superphosphate) fertilizer and in additional fertilizing. Especially effective on alkaline and neutral soils. Double superphosphate is a concentrated water-soluble fertilizer obtained by the decomposition of fresh natural phosphate with phosphoric acid. In appearance, it is a granular product of gray color of various shades - from light gray, almost white, to dark gray, - containing a certain amount of a liquid phase.
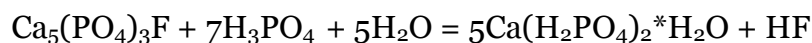
The main component of double superphosphate is monocalcium phosphate monohydrate $Ca(H_2PO_4)_2*H_2O$. Monocalcium phosphate is the most concentrated water-soluble chlorine-free phosphorus-potassium fertilizer with a total nutrient content of 86%. The high content of phosphorus and potassium ensures its efficient use on soils with low fertility.

Table 1 Monocalcium phosphate composition

Mass fraction of assimilated phosphates in terms of P_2O_5, %, not less	52
Mass fraction of potassium in terms of K_2O,%, not less	34
Mass fraction of water-insoluble residue in terms of dry matter,%	0.1
Mass fraction of water,%, no more	0.5
Mass fraction of chlorine,%	0.003

Double superphosphate is distinguished by a small content of other components formed from impurities of phosphoric acid and natural phosphates (calcium sulfate, iron and aluminum phosphates, monomagnesium phosphate, silicofluorides, as well as unreacted phosphate, dicalcium phosphate, free phosphoric acid and free moisture).

During the chemism of the process of decomposition of phosphates by phosphoric acid, the following main reactions occur:

$$Ca_5(PO_4)_3F + 7H_3PO_4 + 5H_2O = 5Ca(H_2PO_4)_2*H_2O + HF$$
$$CaCO_3 + 2H_3PO_4 = Ca(H_2PO_4)_2*H_2O + CO_2$$
$$(Ca, Mg)CO_3 + 4H_3PO_4 = Ca(H_2PO_4)_2*H_2O + Mg(H_2PO_4)_2*H_2O\ 2CO_2$$

Iron and aluminum oxides form medium phosphates:

$$(Al, Fe)_2O_3 + 2H_3PO_4 + H_2O = 2(Al, Fe)PO_4*2H_2O$$

Medium phosphates of aluminum and iron have a negative temperature coefficient of solubility: with an increase in temperature, their crystallization is accelerated. Amorphous $AlPO_4*2H_2O$ slowly transforms into complex salts $CaAlH(PO_4)_2*6H_2O$, $CaAl_6H_4(PO_4)_8*2H_2O$ and $CaAl_6H_4(PO_4)_8*2H_2O$. Amorphous phosphate $FePO_4*H_2O$ is formed only with an excess of Fe_2O_3 in the liquid phase.

Most often, when pH=2 is reached, the reaction proceeds with the formation of $CaFe_2H_4(PO_4)_4*5H_2O$.

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The released HF interacts with silicon dioxide and form H_2SiF_6 and SiF_4:$4HF + SiO_2 = SiF_4 + 2H_2O$
$SiF_4 + 2HF = H_2SiF_6$

Fluorosilicic acid is converted to calcium, sodium and potassium silicofluorides, and silicon tetrafluoride is partially released in a gaseous state. Moreover, the degree of its release increases with increasing temperature and concentration of P_2O_5 in the liquid phase.

The main reactions are accompanied by an increase in temperature and, consequently, in the partial pressure of SiF_4 vapors. During drying at temperatures above 70 °C, calcium silicofluoride decomposes according to the reaction:

$CaSiF_6(solid) + 2H^+(liquid) \rightarrow H_2SiF_6(liquid) + Ca^{2+}(liquid) \rightarrow SiF_4(gas) + +2H^+(liquid)$
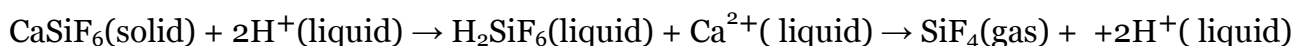
Aluminum ion that enters the solution as a result of the decomposition of by-product minerals of phosphate ores (nepheline, glauconite, and clays) interacts with F^- and SiF_6^{2-} ions to form stronger $[AlF]^{2+}$ complexes than similar silicon compounds.

Their formation delays the release of fluoride compounds into the gas phase.

The stoichiometric norm of H_3PO_4 (n) per 100 mass parts of phosphorite was calculated by the formula:

$$\frac{(\frac{2a}{56.1} + \frac{2b}{40.3} + \frac{2c}{159.7} + \frac{2d}{102} + \frac{2e}{142}) \times 100}{[H^+]a, b, c, d}\%;$$

where e is content in phosphate, respectively CaO, MgO, Fe_2O_3, Al_2O_3 и P_2O_5,

56.1; 40.3; 159.7; 102; 142 are mass of 1 mol of the corresponding components; $[H^+]$ is concentration of hydrogen ions in phosphoric acid.

When decomposing apatite concentrate, the rate of phosphoric acid was calculated using the formula:
where:-

$$n = \frac{f \times 2.33 P_2O_5(п)}{P_2O_5(св об)}$$

f is accepted fraction of stoichiometric norm;

$P_2O_5(ап)$ is content of P_2O_5 in apatite concentrate, %;

$P_2O_5(fr)$ - content of free P_2O_5 in phosphoric acid, %;

2,33 is need for P_2O_5 acid, mass. h for 1 mass. h. P_5O_5 in apatite

The addition to phosphoric acid of such strong acids as nitric, hydrochloric, hydrofluorosilicic and sulfuric acids, or their mixtures, accelerates the phosphate decomposition of phosphates. When phosphate is decomposed by a mixture of acids, the total rate of acids should be determined in terms

of $^nP_2O_5$ or $^nH_3P\ O_4$. The difference between the total rate not exceeding stoichiometric (in terms of phosphoric acid) and the rate of phosphoric acid is the fraction of sulfuric acid expressed in $^nP_2O_5$ or $^nH\ P\ O$

To convert to the norm of sulfuric acid, it is necessary to take into account that 7 mol of phosphoric acid is consumed for the decomposition of 1 mol of apatite, and half of sulfuric acid:

$$2Ca_5(PO_4)_3F + 7H_2SO_4 + 6{,}5H_2O = 3Ca(H_2PO_4)_2*H_2O + 7CaSO_4*0{,}5H_2O + HF$$
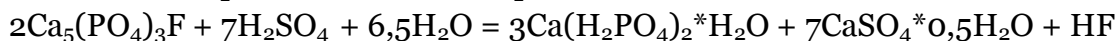
With a very large excess of phosphoric acid (400-500%), fluorapatite can be practically completely decomposed. Usually take 105-110% of the acid from its stoichiometric amount. An increase in the norm of phosphoric acid over the optimal one insignificantly increases the degree of decomposition of phosphate, but significantly worsens the physical properties of double superphosphate and increases the content of free acidity in it.

The change in the degree of decomposition of phosphate over time in the thickening pulp is determined by the combined effect of the activity of hydrogen ions in the solution and the properties of the crystallizing solid phase. With an increase in the acid concentration from 13.6 to 53.6%, the decomposition of the apatite concentrate by the stoichiometric norm of phosphoric acid at 20 °C is accelerated. A further increase in the acid concentration leads to a slowdown in this process.

Moreover, during the first 10-20 minutes, the degree of decomposition depends little on the acid rate. An increase in the rate of acid makes it possible to slightly increase the rate of the process over a longer period of time, however, an increase in the degree of decomposition of phosphate is disproportionate to an increase in the rate of acid due to its incomplete use. With an increase in the rate of acid, the content of free acidity in superphosphate increases, which will require further neutralization to improve the quality of the finished product.

At a low acid consumption rate, the quality of the double superphosphate deteriorates due to the low degree of phosphate degradation. An increase in the acid rate above 110% stoichiometric does not give a noticeable improvement in the quality of the product, but it can lead to the fact that the superphosphate in the chamber will not set and flow out of it.

The degree of decomposition of phosphate increases significantly with an increase in temperature from 20 to 40 °C. A further increase in temperature to 70-80 °C leads to a slight increase in the degree of decomposition. This is due to the fact that at temperatures above 40 °C, already after a very short period of time from the start of the reaction, a significant amount of monocalcium phosphate crystals are released, which create resistance to acid diffusion, as a result of which the rate of the process at temperatures above 40 °C is determined by the diffusion resistance of the solid crust of the reaction product, which has little or no temperature dependence. At temperatures below 40 °C, the degree of decomposition of apatite is noticeably reduced, therefore, operation with cold acid leads to disturbances in the technological regime.

Amount of fluoride compounds released into the gas phase can be estimated from the equilibrium vapor pressure of HF and SiF_4 above H_3PO_4 - H_2SiF_6 - H_2O system. Conditions promoting increase in the

partial pressure of SiF_4 and HF above solutions at equilibrium should favor increase in the fluorine yield into the gas phase under production conditions.

Presence of impurities significantly distorts the process of release of fluoride compounds into the gas phase. The binding of fluorine into strong complexes with aluminum and iron ions, the formation of compounds such as fluorides of aluminum, magnesium, calcium, as well as salts of fluorosilicic acid ($CaSiF_6$, Na_2SiF_6, K_2SiF_6, $MgSiF_6$, etc.) helps to reduce the release of fluoride compounds into the gas phase. Therefore, the degree of fluorine release into the gas phase under production conditions does not exceed 50-55%.

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