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БАЯНДАМАЛАРЫ

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НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН

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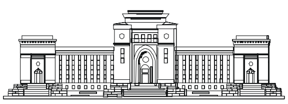

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INVESTIGATION OF ENDOTHERMIC PROPERTIES OF INDUSTRIAL WASTES

Abstract. At the present time the applied fire-extinguishing standard compositions are very expensive and therefore are perspective investigation of fire-extinguishing properties of the pulverized industrial waste. This is connected with their cheapness, low expenses for initial reworking and possibility of their utilization.

At the analysis carrying out of patent and scientific-technical literatures on the question of presence the fire-extinguishing powders it was identified that basic powder components are in non-combustible metallurgic waste.

The searching of effective flash suppressors with waste using is rational in the direction of complex compositions development having low temperatures of endothermic processes of melting, decomposition or evaporation with inert gases formation, but for easy-flammable powders are many-component eutectic mixtures. There are investigated more than 250 minerals and mountain rocks in order to identify endothermic effects. The following ones have endothermic effects in the field of low temperatures: $CaCO_3$ • MgCO.3- dolomite; $CaCO_3$ - calcite, aragonite; $MgC0_3$- magnesite; Ca (Mg, Fe) $(C0_3)_2$ - ankerite; Na_2 $S0_4$• $10H_2O$ - mirabolite; A1 $(OH)_3$ + $A1_4[Si_40|o]$ • $(OH)_8$ –boxite.

Keywords: investigation of endothermal processes, industrial wastes, fire-extinguishing compositions, fire-extinguishing powders, endothermic effects, powder compositions, explosion suppressing compounds, ammophos, derivatograph, thermograms.

Introduction. Analyzing scientific and technical literature of the authors [1-10], it can be concluded that scientists came to a common opinion only in the following: the most effective are substances that are capable of heating to endothermic transformations, that is, volatile, or light-boiling, or easily decomposable. However, nothing is reported about the indices of this process, although it follows logically that the lower the temperature of the beginning of the process and the greater the thermal effect of the endothermic reactions, the better the powder is.

Therefore, for the first time, we propose a technique for the experimental study of the endothermic properties of powders, which determines the temperature range and the thermal effect of the powder, according to which the relative phlegmatizing ability of the compositions can be estimated and predicted.

The essence of the method is to study the processes by the thermal effect accompanying them and change in relative temperature difference at a linear heating rate of the sample under study and the model sample that does not undergo any internal transformations during heating.

When heated, a sample has a lower temperature in comparison with the temperature of the model sample, as a result of endothermic transformations in the region of critical temperatures. During this period of heating, the heat input to the system is less than the heat removal to the environment, so the temperature of the sample is significantly reduced, and the occurrence of combustion becomes impossible.

The moment of a significant decrease in the heating rate and the jump in the differential temperature curve is taken as the start of the endothermic process. The temperature taken at this point on the thermogram is considered as the cooling start temperature.

The method allows us to follow other changes in the sample during heating, accompanied by negative or positive thermal effects (melting, thermal decomposition, etc.), which is very important in the study of multicomponent mixtures.

Based on the results of the analytical review in forecasting, searching for and developing new effective explosion suppressing compounds, the following factors should be taken into account:

1. Powder compositions intended for effective suppression of ignition and explosion must be made from non-deficient domestic raw materials by simple technology.

2. The search for effective flame arresters using wastes is expedient in the development of complex compositions having low temperatures of endothermic processes of melting, decomposition or evaporation with the formation of inert gases; as for the production of low-melting powders, the search should be conducted among multicomponent eutectic mixtures.

Therefore, in the first place, a qualitative analysis of the endothermic capacity of natural rocks, minerals and wastes of production processes should be carried out as the cheapest, non-deficit and not requiring sophisticated production technology (mainly grinding). Analytical studies [11,12] of more than 250 minerals and rocks were carried out by standard albums of thermograms of their diagnostics. As a result, it was found that more than half of them have endothermic effects, but many also have exothermic effects, or they are rare or toxic. Therefore, thanks to endothermic effects in the low temperature region, the following minerals and rocks can be promising:

1. Dolomite $CaCO_3 \cdot MgCO_3$
2. Calcite, aragonite $CaCO_3$
3. Magnesite $MgC0_3$
4. Ankerite $Ca\,(Mg, Fe)\,(C0_3)_2$
5. Mirabilite $Na_2\,S0_4 \cdot 10\,H_2O$
6. Bauxite $A1\,(OH)_3 + A1_4[Si_4\,O_{10}] \cdot (OH)_8$

Analytical forecasting of fire-extinguishing ability of minerals and rocks. Taking into account the above conditions, a preference can be given to carbonates (due to the range and magnitude of the endothermic effect), crystalline hydrates, soda and gypsum (due to low start temperature). Hydroxides have less effect. From the pulverized waste, the following can be suitable:

- powder of iron oxide and sludge of waste streams of rolling shops, containing calcium and iron chlorides;
- dust of dolomite, limestone and lime from limestone burning shops;
- blast-furnace slag, consisting of calcium, magnesium and aluminum silicates.

During heating, chemical reactions and physical transformations occur, accompanied by thermal effects with the release or absorption of heat. Heats of formation (decomposition) of a wide range of compounds are obtained as average values calculated from experimental thermodynamic data for a large number of compounds of this series.

For the purpose of predicting the fire-extinguishing properties of the powder, a technique is proposed for calculating the energy of cooling the flame [13] (dissociation energy) ΔH□298 on the basis of the Hess thermodynamic law:

$$\Delta H\square_{298(\text{reaction})} = \Sigma\Delta H\square_{298(\textit{prod})} - \Sigma\Delta H\square_{298(\textit{initial})} \quad (1)$$

where $\Sigma\Delta H\square_{298(prod)}$ is the sum of the heats of formation of final products, kJ/mol; $\Sigma\Delta H\square_{298(initial)}$ is the sum of the heats of formation of initial substances, kJ / mol; $\Delta H\square_{298(reaction)}$ is the thermal effect of chemical reaction under standard conditions, kJ/mol.

The heats of the formation of substances are taken from the reference literature [14]. From the calculated decomposition reactions (Table 1) of standard fire extinguishing powders and wastes, it follows that the efficiency of soda is much less than that of lime, but it is impossible to make an unambiguous conclusion, since the temperature of the beginning of the process is not taken into account. Therefore, there is a need in experimental studies, for which a standard differential-thermogravimetric method was used on the derivatograph of the Paulik system.

Synchronous automatic recording of changes when the substance is heated on a thermogram allows us to judge the nature and intensity of thermal effects, and the temperatures at which effects occur.

The endothermic effect is the deviation of the DTA curve down to the abscissa axis, the exothermic effect is upward. The change in the weight of the sample during heating is recorded, which makes it possible to record the gasification of the sample.

Table 1- Energies of powders dissociation

Substance	Reaction of powder decomposition	$\Delta H\square_{298}$, kJ/mol	Temperature ranges of the cooling effect, °C
Soda	$NaHC0_3$= NaOH+CO_2	96	190-450, 780-850
Dolomite	$CaCO_3$ = CaO+CO_2	177,4	430-640, 750-920
	$MgCO_3$= MgO+ CO_2	101,5	
Lime	$Ca(OH)_2$=CaO+H_2O	108,2	430-580, 720-830
Ammophos	$NH_4H_2PO_4$=NH_3+1.5H_2O+0.5P_2O_5	61,2	190-430
	$(NH_4)_2HPO_4$|=4NH_3+3H_20+P_2O_5	168,8	
Carbamide	CH_3N_2OH=CO+N_2+2H_2	870	120-130

The main component of highly effective extinguishing powders, such as PF, P-2AP, P-4AP, R-P-24 is ammophos, consisting of phosphate-ammonium salts.

Based on the reference thermodynamic data on the heats of formation of , H_3PO_4, HPO_3, H_2O, P_2O_5, the binding energies between the NH, O-H, P-O, P = O atoms and the enthalpy of ammophos containing compounds of mono- and diammonium- phosphates such as $NH_4H_2PO_4$ and $(NH_4)_2HPO_4$ are calculated.

As follows from the calculations, the decomposition reaction of ammophos including ammonium dihydrogenphosphate and ammonium hydrogen phosphate proceeds with heat absorption (endothermic); and 584.85 kJ is expended per 1 kg of ammophos.

Thermodynamic calculation of decomposition reactions of dolomite and lime dust also comes with an endothermic process of decomposition and destructuring, which indicates the possibility of their use in fire extinguishing compositions. The calculation procedure is given in Appendix A.

The calculated data obtained are in good agreement with the thermographic studies of pulverized materials conducted with DTA method. When the dolomite dust is heated (Fig. 1 a), two endothermic effects are observed on the thermogram: the first - in the temperature range from 430°C to 640°C and the second - from 750°C to 920°C. It can be explained by the thermochemical transformations taking place. Judging by the presence in the sample of up to 45% loss on ignition, i.e. gaseous substances released when the sample is heated, the dust contains carbonates and hydroxides of calcium and magnesium. The temperatures of their decomposition according to the reference data are (°C): for $CaCO_3$ - 825; $MgCO_3 \cdot 3H_2O$-165; Mg $(OH)_2$ - 200; $MgCO_3$ - more than 350; Ca $(OH)_2$- 580.

When lime is heated, the first endothermic effect is determined by the dissociation of calcium hydroxide in the reaction (3.2) with the participation of decomposition of impurities of magnesium compounds:

$$Ca(OH)_2 = CaO + H_2O \qquad (2)$$

The beginning of the first endothermic peak occurs at a temperature of 430°C and corresponds to the onset of dissociation of magnesium carbonates, and the second peak occurs at 720°C dissociation of calcium carbonates. Decomposition temperatures are close to the reference data. Loss on ignition of lime is 16%.

The second peak in reaction (3.3) is explained by the dissociation of the incompletely burned calcined lime:

$$CaCO_3 = CaO + CO_2 \tag{3}$$

and decomposition of calcium hydroxide. When the dolomite samples are heated, the first endoeffect is due to the decomposition of magnesium carbonate, the second endoeffect is explained by the onset of the decomposition of calcium carbonate.

Oxides act upon heating as inerts and their antipyrogenic properties are weak.

However, the effect of phlegmatization by inert substances increases with the use of substances with endothermic melting processes [15]. They include the commonly used magnesium, potassium, and sodium chlorides with temperatures of 700°C to 800°C. Hydrochloric acid effluent of the plant with a volume of about 1.3 million cubic meters per year, neutralized with a 5% solution of lime, is discharged into the reservoir. The resulting calcium chloride is widely known as an effective antipyrogen.

The effect of carbonates and hydroxides, in addition to physical absorption of heat by analogy with oxides, is due to the chemical cooling of the reaction zone, i.e. endothermic effect. Moreover, under these reactions, water vapor and carbon dioxide are released into the atmosphere, which serve as effective flame retardants. This indicates the possibility of using dolomite dust and lime dust in fire extinguishing compositions.

To compare and evaluate the reliability of the indications obtained at the installation of South Kazakhstan State University (SKSU), the similar samples were studied on the derivatograph of the Paulik system. The heating rate was 10 deg / min, pure aluminum oxide was used as a reference substance. Parallel studies on the derivatograph gave similar results, thermal effects are observed in the same sequence, but they have smoother peaks shifted to the lower temperatures zone. Comparative results of thermogravimetric analysis of pulverized industrial waste and fire extinguishing powder are given in Table 2.

Table 2- Results of thermogravimetric analysis of industrial pulverized wastes

Sample	Ratio %	Installation of SKSU			Derivatograph		
		Temperature,^{0} C			Temperature,^{0} C		
		T_b	T_e	max ДТА	T_b	T_e	max ДТА
Powder P-2AP	100	200	480	430	190	430	350
Dolomite	100	510, 800	680,960	520,900	430,750	640,920	490,880
Lime	100	500,780	590,880	590,830	430,720	580,830	500,780
Powder P-2AP	100	180	450	400	150	440	390
Dolomite	100	480,800	680,960	500,890	420,780	640,900	500,890
Lime	100	490,780	580,870	580,820	420,730	570,820	490,780
Powder P-2AP	100	190	450	420	180	430	420
Dolomite	100	500,780	690,950	520,890	430,720	630,920	490,890
Lime		500,770	590,890	580,830	420,720	580,820	500,760

The study of standard extinguishing powders (see Figures 1) showed that the endothermic effects of soda and ammophos are much greater than that of gypsum and sodium chloride.

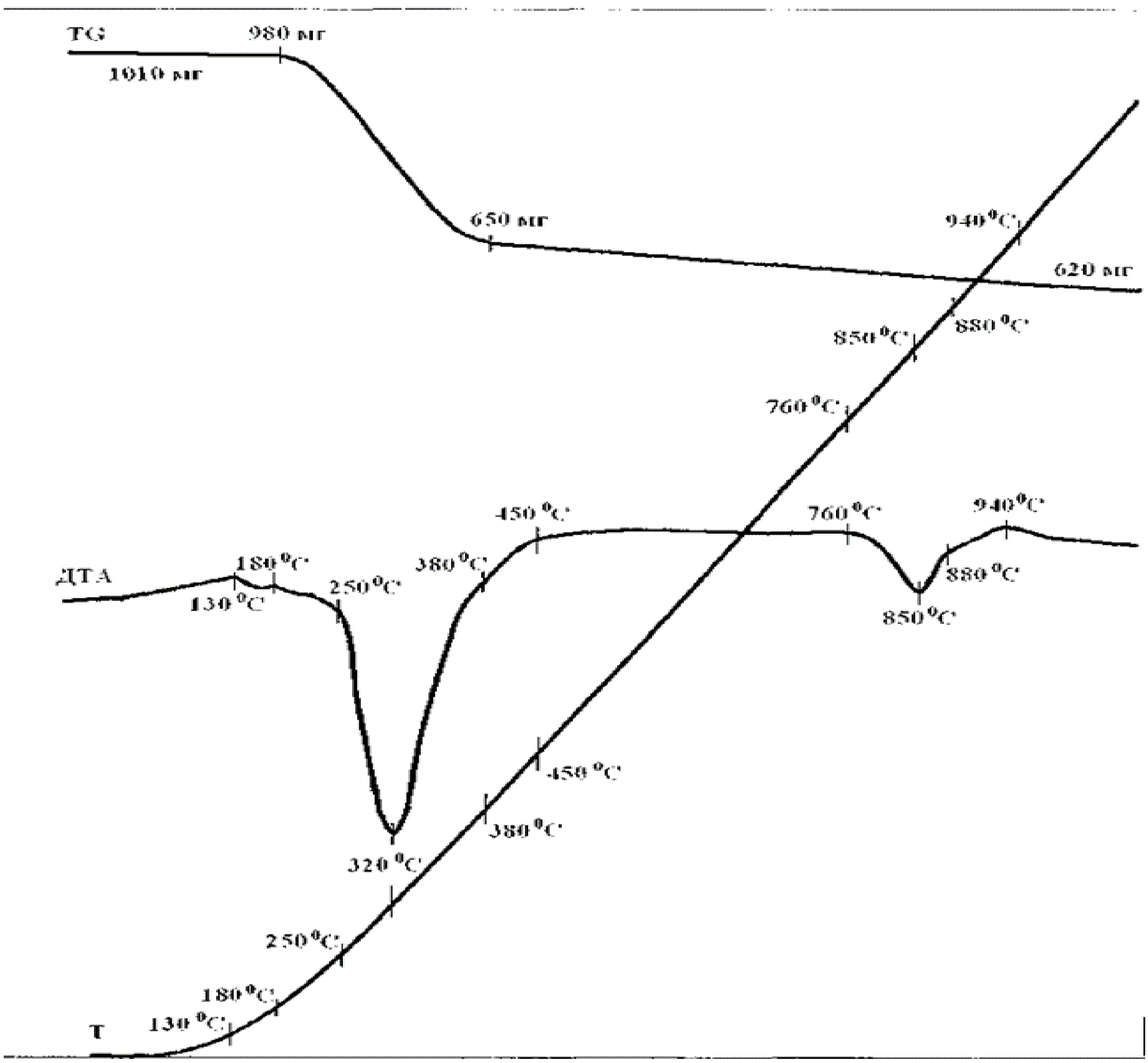

Figure 1 - The thermogram of soda decomposition

In thermograms of pulverized wastes (see Figures 2), endothermic effects of lime and dolomite are significant, which indicates their decomposition with the formation of oxides and the release of carbon dioxide. This is confirmed by the loss of their weight. The endothermic effects of blast furnace slag and iron oxide are minimal. By the way, all thermograms are similar to standard curves.

Weight loss curves TG:

1'- lime; 2'- dolomite; 3'- blast furnace slag; 4'- iron oxide

DTA curves:

1 - lime; 2 - dolomite; 3 - blast furnace slag; 4 - iron oxide

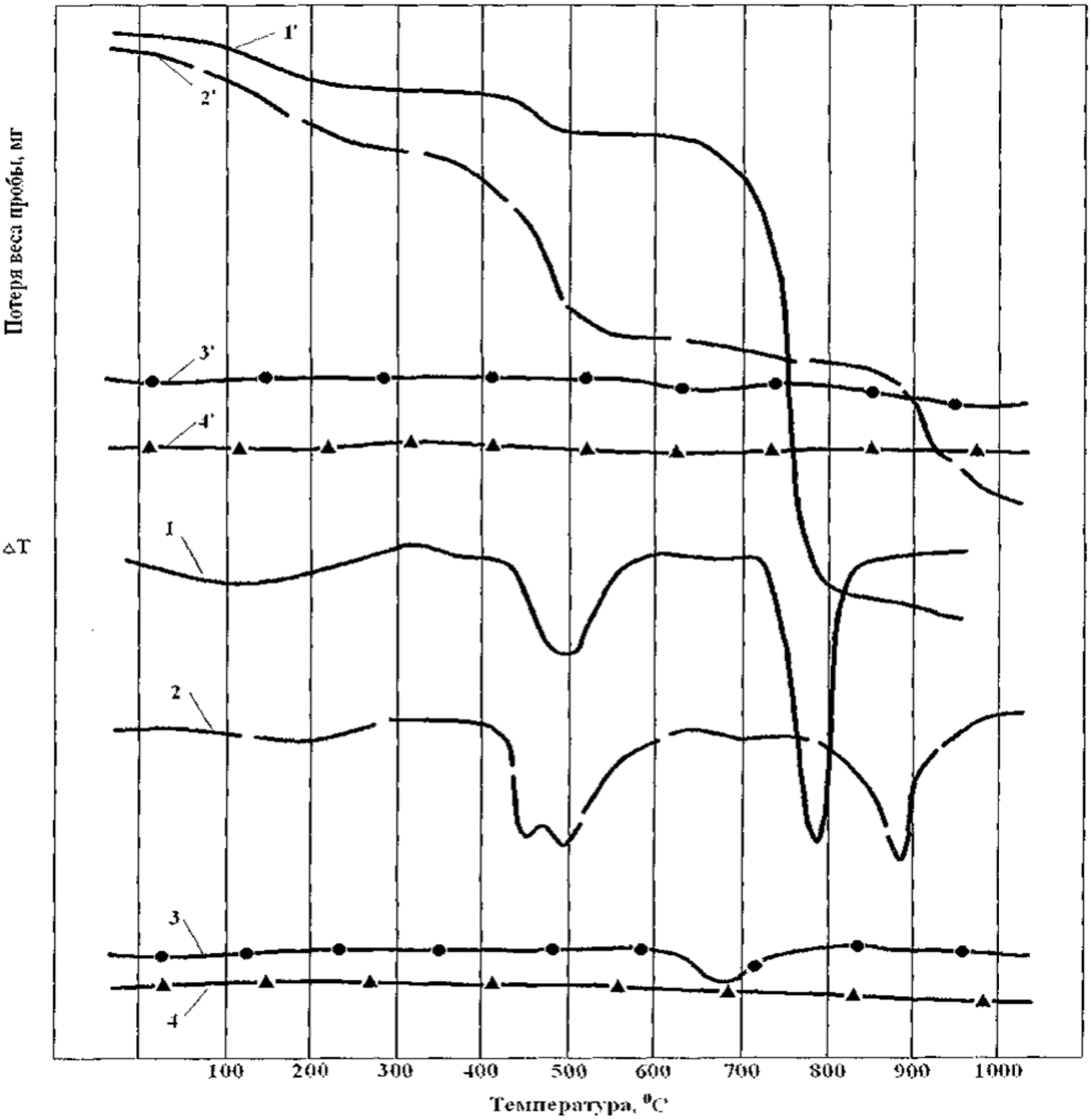

Figure 2 - Thermograms of dispersed metallurgical wastes

It can be seen that the first losses of water and ammonia are observed at the values of temperature up to 180°C. The endothermic effect at 190°C refers to the process of transition of diammonium phosphate to monoammonium phosphate and the formation of ammonium pyrophosphate from monoammonium phosphate. After 200°C, the polymerization of monomeric phosphates begins in the melt with the endo-effect at 240 ° C at which further loss of ammonia and water occurs. The changes at temperatures of 440, 550 and 715 ° C indicate further polymerization and the preparation of compounds that form viscous films on the smoldering surface that hinder the access of oxygen to the burning surface. The total loss of ammophos mass is more than 70% [16-18].

Based on the experiments results and taking into account the cheapness, dispersity and environmental safety of raw materials, we can conclude that dolomite, hydrated lime and expired fire extinguishing powder are promising for further research.

Conclusions. It is shown that the basis for mass-produced fire extinguishing powders, i.e. the most effective flame arresters currently used are salts: phosphates, carbonates and chlorides of ammonium and alkaline earth metals.

The effectiveness of these powders is due to the endothermic processes of decomposition and destruction that occur during heating, which are cooling the combustion zone, which is confirmed by differential-thermographic analysis of minerals and rocks. A similar forecast is also promising for hydrates and hydroxides.

It is established that among metallurgical wastes the basis for the production of effective fire extinguishing powders can be: dust and dispersed waste of dolomite and hydrated lime of limestone burning shops containing calcium and magnesium carbonates, and calcium hydroxide.

It has been established that the efficiency of powders can be quantitatively determined by complex consideration of the endothermic effect (the area of the endoeffect on the thermogram), the initial temperature and the decomposition rate of the substance.

It is shown that from the point of view of low cost of raw materials, ecological safety and simplicity of technology, disperse waste (dust) of dolomite and slaked lime of limestone burning shops is promising for the development of complex powder compositions.

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ӨНДІРІСТІК ҚАЛДЫҚТАРДЫҢ ЭДОТЕРМИЯЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

Аннотация. Қазіргі уақытта өрт сөндіруде қолданылатын стандартты қоспалар өте қымбат келеді. Сондықтан ұсақталған өнеркәсіптік қалдықтардың өрт сөндіру қасиеттерін зерттеудің болашағы зор. Бұл олардың құнының төмен боуымен, сондай-ақ бастапқы өңдеуге жұмсалатын шығындардың аздығы мен оларды кәдеге жаратып пайдалану мүмкіндіктерімен байланысты.

Өрт сөндіру ұнтақтарының құрамы туралы патенттік және ғылыми әдебиеттерді талдау кезінде негізгі ұнтақ компоненттері жанбайтын металлургиялық қалдықтардан тұратындығы анықталды.

Қалдықтарды пайдалану арқылы жарылыстарды болдырмаудың тиімді құралдарын іздестіру инертті газдардың қалыптасуымен эндотермиялық процестердің балқу, ыдырау немесе булану үрдістерінің төмен температуралы, алайда тез тұтанатын ұнтақтар үшін көп компонентті эвтектикалық қоспалар болып табылатын кешенді қоспаларды дамыту бағытында ұтымды.

Эндотермиялық әсерлерді анықтау үшін 250-ден астам минералдар мен тау жыныстары зерттелген. Төмен температурадағы эндотермиялық әсерлер мыналардан тұрады: CaCOз • MgCO.3- доломит; CaCOз - кальцит, арагонит; MgCO3-магнезит; Ca (Mg, Fe) (C03) 2 - анкерит; Na2S04 • 10H2O - мираболит; A1 (OH) 3 + A14 [Si40 | o] • (OH) 8 – боксит.

Түйін сөздер: эндотермиялық процестерді зерттеу, өнеркәсіптік қалдықтар, өрт сөндіру құрамы, өрт сөндіргіш ұнтақтары, эндотермиялық әсерлер, ұнтақ құрамы, жарылғыштықты басатын құрамдар, амофос, дериватограф, термограммалар.

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ИССЛЕДОВАНИЕ ЭНДОТЕРМИЧЕСКИХ СВОЙСТВ ПРОМЫШЛЕННЫХ ОТХОДОВ

Аннотация. В настоящее время применяемые стандартные композиции для пожаротушения очень дороги и поэтому являются перспективным исследованием свойств пожаротушения измельченных промышленных отходов. Это связано с их дешевизной, низкими затратами на первичную переработку и возможностью их утилизации.

При проведении анализа патентной и научно-технической литературы по вопросу о наличии огнетушащих порошков было выявлено, что основные порошковые компоненты находятся в негорючих металлургических отходах.

Поиск эффективных средств подавления взрывов с использованием отходов является рациональным в направлении разработки сложных композиций, имеющих низкие температуры эндотермических процессов плавления, разложения или испарения с образованием инертных газов, но для легковоспламеняющихся порошков представляют собой многокомпонентные эвтектические смеси.

Для выявления эндотермических эффектов исследовано более 250 минералов и горных пород. Эндотермические эффекты в области низких температур имеют следующие: CaCOз • MgCO.3- доломит; CaCOз - кальцит, арагонит; MgCO3-магнезит; Ca (Mg, Fe) (C03) 2 - анкерит; Na2S04 • 10H2O - мираболит; A1 (OH) 3 + A14 [Si40 | o] • (OH) 8 – боксит.

Ключевые слова: исследование эндотермических процессов, промышленные отходы, огнетушащие составы, огнетушащие порошки, эндотермические эффекты, порошковые составы, взрывоподавляющие составы, аммофос, дериватограф, термограммы.

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