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STUDY OF THE TECHNOLOGY FOR THE PRODUCTION OF INDIVIDUAL URINE-FORMALDEHYDE COMPOUNDS OF MULTIFUNCTIONAL ACTION ON THE BASIS OF UFC AND ZINC ACETATE IN SPRAY DRYER

Abstract. This article presents studies of the process of obtaining low-molecular urea-formaldehyde compounds, drugs of multifunctional action on the basis of low-molecular urea-formaldehyde compounds and zinc acetate in a spray dryer. Optimum technological parameters of their production are determined. The temperature of the heat carrier is 250-30 °C, the temperature in the reaction zone is 70-90 °C, the moisture content of the obtained products is 1-4%.

Key words: urea formaldehyde compounds, zinc acetate, drugs of multifunctional action, drying process, phase composition changes, polycondensation process, spray.

Introduction. Urea-formaldehyde compounds and fertilizers based on them are obtained in a drying drum, a drum-granulator-dryer, under fluidized bed conditions [1-3]. Obtaining of these fertilizers in the form of a powder for low-tonnage chemistry would become great practical tool for the field of chemistry and industrial chemistry.

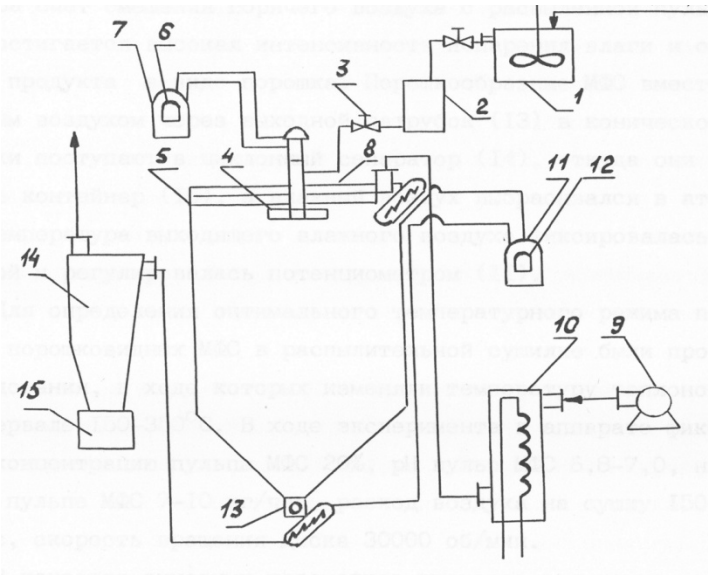
The task of the search was study the process of obtaining low-molecular urea-formaldehyde compounds (UFC) – monomethylolurea, dimethylolurea, methylene-diurea and multifunctional action compounds such as a zinc acetate in powder form.

EXPERIMENT

A pilot plant the main unit of which is a spray dryer is represented in figure. The pulps of UFC and drugs of multifunctional action based on them and zinc acetate were obtained in a tank heated reactor with a stirrer (1), where water, UFC and zinc acetate were supplied based on the calculation of 20% pulp. By feeding the heating steam into the jacket of the reactor, the pulp temperature was maintained at 40-45 °C, at which the pulp was well mobile.

As drugs of multifunctional action based on UFC and zinc acetate, double compounds obtained on the basis of monomethylolurea and zinc acetate, dimethylolurea and zinc acetate obtained at molar ratios of 1: 1, as well as methylene-diurea of 4 molecules and 1 molecule of zinc acetate were used.

Further pulps from the tank (1) was fed to the pressure vessel (2), from where it was dosed to the central disk of the spray dryer (5) by the means of a calibrated valve (3). The rotational speed of the disk was 30,000 rpm and was



Experimental installation for the production of urea-formaldehyde compounds and compounds of multifunctional action containing zinc acetate:
1 - capacity for the preparation of UFC pulps and drugs of multifunctional action based on them and zinc acetate, 2 - pressure vessel, 3 - valve, 4 - central disk, 5 - spray dryer, 6 - regulator, 7 - amperemeter, 8 - dryers, 9 - fan, 10 - (calorifer) heater, 11 - potentiometer, 12 - temperature controller, 13 - outlet, 14 - cyclone separator, 15 container

varied by a regulator (6), the control was performed by means of a calibrated ampermeter (7). The pulp was sprayed into the apparatus with the aid of a disk to a foggy state. To dry the pulp through the dryers (8) located in the ceiling of the dryer, a coolant was introduced- hot air with a temperature of 150-350 °C, fed to the unit by a fan (9) through the heater (10). The air temperature was fixed by a potentiometer (11) and regulated by a temperature regulator (12).

By mixing hot air with pulverized pulps, a high intensity of moisture evaporation and the formation of the product in the form of a powder are achieved. Powdered UFC and compounds of multifunctional action together with moist air through the outlet pipe (13) in the conical bottom of the dryer enter the cyclone separator (14), from which they were poured into the container (15), and the moist air entered the stage of dust and gas cleaning. The temperature of the moist humid air was fixed by a thermo couple and controlled by a potentiometer (11).

RESULTS AND DISCUSSION

To determine the optimum temperature regime for the preparation of powdered preparations in a spray dryer, studies were performed in which the temperature of the coolant was varied in the range 150-350 °C. In the framework of the experiment, the concentration of UFC pulps and compounds of multifunctional action on their basis and zinc acetate was 20%, pH 6.8-7.0, loading on the pulp 7-

10 kg/h, air consumption for drying 150-200 m^3/h, the rotational speed of the disk is 30000 rpm.

When a coolant with a temperature of 150-200 °C was applied to the spray dryer, caking occurred in the apparatus due to insufficient drying of the compounds having a temperature of 55-60 °C. When the temperature of the drying agent is increased to 250-300 °C, agglomeration and clumping of products are eliminated, the temperature of the material in the apparatus increases to 70-90 °C, which makes it possible to obtain in the spray dryer a well-flowing powdery UFC and compounds of multifunctional action based on them and zinc acetate. When the coolant is supplied to the apparatus with a higher temperature of 300-350 °C, the temperature of the material in the apparatus increases to 95-110 °C, which leads to fusion of mono- and dimethylolurea, to the partial decomposition of UFC and, as a result, to increased losses of formaldehyde to the gas phase.

Thus, the temperature of the coolant when powdered preparations based on UFC is obtained in the spray dryer must not exceed 250-300 °C at the inlet to the apparatus, in which the optimum temperature regime of the process and the minimal thermal decomposition of UFC are maintained in the dryer.

Table 1 displays the composition of the drying product of monomethylolurea pulp and the quantitative characteristics of gas phase to be related to formaldehyde and product dust. It was found that at a pH of 7.0 and a product temperature of 70-90 °C, condensation of the product is excluded and a product of stable composition can be obtained.

Table 1 – Composition of the drying product of the monomethylolurea pulp in a spray dryer and the composition and concentration of gas phase

Parameter		Composition of the product, mass %			Composition and concentration of gas phase, mg/m^3	
pH	Product temperature, °C	Monomethylolurea	Condensation product	Free urea	Formaldehyde	Dust products
7,0	70	97,5	0	2,5	100	200
	90	97,5	0	2,5	100	300
	95	97,5	1,0	2,5	110	400
6,8	70	97,5	0	2,5	100	250
	90	97,5	0,5	2,5	110	350
	95	94,0	3,5	2,5	120	500

Characteristically, with an increase in the temperature of monomethylolurea in the apparatus to 95 °C, a minimal change in its composition occurs, the content of the condensation product is 1.0%, whereas in the production of solid monomethylolurea by the thin layer method, under laboratory conditions, the mass fraction of the condensation product is 3.0-5,0%. This is apparently due to the fact that the drying of UFC solutions in the spray dryer is rapid, within a second they

lose moisture and settle in the form of fine powder particles, this almost instantaneous dehydration of the product leads to a slowdown of the condensation process. The decrease in pH in the supplied monomethylolurea solutions to the apparatus up to 6.8 contributes to the condensation process when drying the product at a temperature of 95 °C, the condensate content is 3.5%.

However, this parameter is 2-3 times less than in case of thin-layer drying of monomethylolurea. This also confirms that rapid dehydration of the product reduces the course of the polycondensation reaction.

In optimal conditions of the technology of production in exhaust gases after spray dryer in the production of powdered monomethylolurea, the formaldehyde concentration is 100 mg/m^3 and the dust of the product is 200-250 mg/m^3, which must be disposed.

In the production of powdered dimethylolurea in a spray dryer, the product of stable composition was obtained at the range temperature from 220 to 370 °C then supplied to coolant apparatus and the product has temperature about 70-90°C and pH 6.8-7.0 (table 2).

Table 2 – Composition of the product at drying of pulp dimethylolurea in a spray dryer and composition of gas phase

Experiment conditions		Composition of the product, mass %			Composition and concentration of the gas phase, mg/m^3	
pH	Product temperature, ^{o}C	Monome-thylolurea	Condensation product	Free urea	Formal-dehyde	Dust products
7,0	70	90,9	0	9,1	150	220
	90	90,9	0	9,1	150	340
	95	88,4	2,0	9,6	160	400
6,8	70	91,9	0	8,1	150	260
	90	90,9	0	9,1	150	370
	95	85,0	4,2	10,8	170	500

An increase in the product temperature in the drier to 95 °C promotes the polycondensation of dimethylolurea, the content of the condensation product is 2.0-4.2%. In the gas phase, after the spray dryer under optimal conditions for the production of dimethylolurea powder, 150 mg/m^3 of formaldehyde and 220-370 mg/m^3 of product dust are contained.

When a powder of methylene-diurea was obtained in a spray dryer at a pH of the pulp of 6.8-7.0, a product of stable composition was obtained in the temperature range 70-95°C, and its condensation was eliminated. In the exhaust gases after the spray dryer, formaldehyde is absent and the product dust contains 220-370 mg/m^3.

Thus, the process of obtaining powdered UFC - mono-, dimethylol-, methylene-diurea in a spray dryer was studied. Optimum technological parameters of the process of drying products with residual moisture content of 1.0-1.5% with a high yield of a stable composition product were determined (table 3).

Table 3 – Optimum technological parameters of the process for the production of powdered urea-formaldehyde compounds in a spray dryer

#	Parameters	Unit	Value
1	Temperature:		
	a) of coolant at the inlet to the spray dryer	^{o}C	250-300
	b) of coolant at the outlet from the dryer	^{o}C	80-90
	c) UFC pulp	^{o}C	40-45
	d) powdered product of MPS in the reaction zone	^{o}C	70-90
2	UFC pulp concentration	%	20
3	UFC pulp load	kg/h	7-10
4	pH of the UFC pulp		6,8-7,0
5	The residence time of the product in the reaction zone	minutes	2-5
6	The moisture content in the powdered product	%	1,0-1,5
7	Air consumption for drying	m^3/h	150-200
8	Productivity	kg/h	2-3

In the process of studies on the preparation of compounds based on low molecular weight UFC and zinc acetate in a spray dryer, the task was also to study the effect of technological process parameters on the composition of finished products, determine the amount and composition of gas emissions after the dryer.

When multifunctional action compounds based on mono- and dimethylolurea and zinc acetate are obtained in the spray dryer in the temperature range of the material in the reaction zone of the apparatus 70-90 ^{o}C and pH 6.5-7.0, hydrolysis and condensation of methylol formaldehyde in the composition of binary salts is excluded and it is possible to obtain powdered compounds of stable composition (table 4). It should be noted that an increase in the temperature of the compounds in the drier to 95 °C results in a relatively small degree of polycondensation of mono- and dimethylolurea in the composition of binary salts, as a result of which the mass fraction of methylol formaldehyde in the product based on monomethylolurea and zinc acetate is reduced to 95.0-98, 5% and the formation of 1,5-5,0% methylene formaldehyde, in the case of the preparation based on dimethylolurea, methylol formaldehyde decreases to 4.5-9.0%. Whereas in the preparation of solid compounds based on mono-, dimethylolurea and zinc acetate under laboratory conditions by drying in thin layers at pH of salts of 6.5-7.0 and a temperature of 95 °C, the mass fraction of methylene formaldehyde in products was 8-10% and 14 -17% respectively. This can be explained by the fact that the drying of pulp compounds based on mono- and dimethylolurea in a spray dryer occurs almost instantaneously, within a fraction of a second they lose moisture and settle in the form of fine powder particles; this rapid dehydration of the products helps to inhibit the condensation process.

Table 4 – The content of methylol and methylene formaldehyde in the pulp drying products of compounds based on mono-, and dimethylolurea, zinc acetate in a spray dryer and the composition of the gas phase

Double connection	Experiment conditions		Mass fraction of formaldehyde in the product,%		Concentration of the gas phase, mg/m^3	
	pH	Product's temperature, ^{o}C	Methylol	Methylene	Formal-dehyde	Product dust
Monome-thylolurea zinc acetate	7,0	70	100,0	0	55	200
		90	100,0	0	55	280
		95	98,5	1,5	70	360
	6,5	70	100,0	0	60	230
		90	100,0	0	60	300
		95	95,0	5,0	80	450
Dimethylolurea zinc acetate	7,0	70	100,0	0	70	240
		90	100,0	0	70	350
		95	95,0	4,5	90	430
	6,5	70	100,0	0	85	250
		90	100,0	0	85	380
		95	91,0	9,0	100	470

In the exhaust gases after the spray dryer for the preparation of powder formulations based on mono- and dimethylolurea, zinc acetate, the concentration of formaldehyde is 55-85 mg/m^3 and the dust of the product is 200-380 mg/m^3, which must be disposed of.

In the preparation of a preparation based on methylene-diurea and zinc acetate in a spray dryer at a pH of 6.5-7.0 in the temperature range of 70-95 °C, hydrolysis of the product and condensation of methylene-diuria in the composition of the double compound does not occur, and conditions for obtaining a preparation of stable composition are provided. In the exhaust gases after the spray dryer in the preparation of the drug based on methylene-diurea, formaldehyde is absent, contains 200-300 mg/m^3 of the product dust. The optimum technological parameters of the drying process of the products were determined, ensuring the preparation of multifunctional action compounds of stable composition based on UFC and zinc acetate (table 5).

Table 5 – Optimum technological parameters of the process for the preparation of powder compounds based on urea-formaldehyde compounds and zinc acetate in a spray dryer

#	Parameters	Unit	Values
1	Temperature:		
	a) of coolant at the inlet to spray dryer	^oC	250-300
	b) coolant at the outlet from the dryer	^oC	80-90
	c) pulp of the preparation	^oC	40-45
	d) a powdered preparation in the reaction zone	^oC	70-90
2	Concentration of pulp	%	20
3	Load on the pulp of the preparation	kg/h	6-10
4	pH of the drug's pulp		6,5-7,0
5	The residence time of the preparation in the reaction zone	minutes	2-5
6	The moisture content	%	2,0-4,0
7	Air consumption for drying	m^3/h	150-200
8	Productivity	kg/h	2-4

Conclusion. Thus, the optimal technological parameters for the preparation of low molecular weight MPS – monomethylolurea, dimethylolurea, methylene-diurea and multifunctional action compounds based on them and zinc acetate in a spray dryer, are obtained in which a qualitative, loose product is obtained: the temperature of the heat carrier is 250-300 ° C, the temperature in the reaction zone of the 70- 90 ^oC, the moisture content of the obtained products is 1-4%. The off-gas contains 55-150 mg/m^3 of formaldehyde and 200-380 mg/m^3 of product dust, which must be disposed.

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Резюме

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ЖЕКЕ МОЧЕВИНО-ФОРМАЛЬДЕГИДТІК ҚОСЫЛЫСТАР МЕН ОЛАРДЫҢ НЕГІЗІНДЕ МЫРЫШ АЦЕТАТЫМЕН ПОЛИФУНКЦИОНАЛДЫ ӘРЕКЕТТІ ПРЕПАРАТТАР АЛУДЫҢ ТЕХНОЛОГИЯСЫН ЗЕРТТЕУ

Мақалада төменмолекулалы мочевино-формальдегидтік қосылыстар мен олардың негізінде мырыш ацетатымен полифункционалды әрекетті препараттарды бүркеуші кептіргіште алу процессінің зерттеулері көрсетілген. Оларды алудың оңтайлы технологиялық параметрлері: жылуұстағыш температурасы 250-300 ^{o}C, реакциялық алаңда температурасы 70-90 ^{o}C, алынған өнімдердің ылғалдылығы 1-4%.

Түйін сөздер: мочевино-формальдегидті қосылыстар, мырыш ацетаты, полифункционалды әрекетті препараттар, кептіру процесі, фазалық құрам өзгерістері, поликонденсация процессі, бүркеуші кептіргіш.

Резюме

Ф. Р. Ермаханова, С. Усманов, Г. Т. Омарова

ИССЛЕДОВАНИЕ ТЕХНОЛОГИИ ПОЛУЧЕНИЯ ИНДИВИДУАЛЬНЫХ МОЧЕВИНО-ФОРМАЛЬДЕГИДНЫХ СОЕДИНЕНИЙ И ПРЕПАРАТОВ ПОЛИФУНКЦИОНАЛЬНОГО ДЕЙСТВИЯ НА ИХ ОСНОВЕ И АЦЕТАТА ЦИНКА В РАСПЫЛИТЕЛЬНОЙ СУШИЛКЕ

Исследован процесс получения низкомолекулярных мочевиноформальдегидных соединений, препаратов полифункционального действия на их основе и ацетата цинка в распылительной сушилке. Определены оптимальные технологические параметры их получения: температура теплоносителя 250-300 ^{o}C, температура в реакционной зоне 70-90 ^{o}C, влагосодержание полученных продуктов 1-4%.

Ключевые слова: мочевино-формальдегидные соединения, ацетат цинка, препараты полифункционального действия, процесс сушки, изменения фазового состава, процесс поликонденсации, распылительная сушилка.