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NEW MODIFICATION OF POTASH FERTILIZER DRYING IN FLUIDIZED BED (FB)

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RESUME

NIIGIPROCHIM (St. Petersbourg) a mis au point une série unifiée d'unités de lit fluidisé pour des débits allant de quelques centaines de kg à 130 ou 150 t/h. Les usines de traitement du minerai de potasse dans l'Oural et en Biélorussie emploient des unités à lit fluidisé comprises dans les séries unifiées NIIGIPROCHIM. La production d'une unité peut atteindre 120-150 t/h, l'humidité initiale étant de 6 à 7 % et l'humidité finale ne dépassant pas 0,05 à 0,1 %.

Les procédés de déshydratation à un seul étage pour la kaïnite ($KCl.MgSO_4.3H_2O$), la carnalite ($KCl.MgCl_2.6H_2O$) et des solutions de chlorure de magnésium ont été étudiés. Dans la première phase, la déshydratation est effectuée pour obtenir 2,5 moles d'eau par mole de $MgCl_2$, le degré d'hydrolyse ne dépassant pas 6 %. Avec la technique du lit fluidisé, le procédé est réalisable en une phase avec les appareils classiques. Pour une déshydratation plus poussée, une nouvelle modification d'appareillage est proposée, utilisant un lit de particules inertes comme par exemple, le corindon. La déshydratation se produit à la surface des particules inertes et le produit déshydraté sous forme finement divisée est entraîné par le courant gazeux pour être séparé dans des cyclones et des filtres. Le temps de séjour du produit dans le lit ne dépasse pas quelques secondes et une déshydratation complète peut être réalisée sans augmenter notablement la concentration en magnésie du produit. La déshydration en lit fluidisé basée sur l'emploi d'un lit d'un matériau inerte permet un traitement efficace du phosphogypse et d'autres matériaux. Par rapport à l'équipement classique de lit fluidisé ou de tambour rotatif, cette modification permet d'augmenter le rendement plusieurs fois, de réduire la consommation d'énergie et de fabriquer une gamme importante de produits.

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Numerous publications have been devoted to the process of drying in fluidized bed. However, up to now there is no general theory of the process that can determine the optimum operational mode of industrial units. Moreover, the available notions about kinetic characteristics of drying in FB, are essentially hindering the process optimization. The development of theory regarding the optimal ways and their use for establishing a new modification of drying in FB had been started by us in the potash industry. Furthermore, this method is successfully used for drying a wide range of various materials such as dry flowing (loose) precipitates, suspensions, crystallohydrates and solutions. The general characteristics of the process relationships has enabled us to unify the design of FB units. NIIGIPROCHIM has designed a unified range of FB units with capacities ranging from hundreds of kilogrammes to 120-150 t/hr.

The first units for drying of potash salts in FB appeared in the former USSR in 1960 s, i.e. 5-10 years earlier than those at the European, American and Canadian potash plants.

A comparison of the major process performances of the units now operating shows considerable advantages of the modification presented by us, thus confirming the new theoretical propositions and engineering designs based on them.

Table 1: Comparison of Performances of Fluidized Beds

Performance Indices	European FB	New method FB
Capacity (t/hour)	150	120-150
Grid surface (m^2)	15	6.5
Air consumption (Nm^3 /hour)	100000	40-45000
Fuel consumption (mazout) (kg/hour)	1400	950-1150
Specific consumption:		
a) air, Nm^3 /T product	6500 - 6600	4000-3600
b) fuel, kg/T product	9.3	7.5-8.5

(Yu. Ya. Kaganovich "Fluidized Bed dehydration on an industrial scale" J. Chimia, 1990)

Table 1 shows the comparison of performances indices of heavy-duty fluidized-bed dryers of European company with the new method. For the comparison we choose the apparatus for potassium chloride drying at practically equal initial humidity 6%.

The substantial reduction of air consumption corresponds to the equivalent reduction of power consumption for gases supplied to the bed and overall dimensions of exhaust gases dedusting apparatus.

The initial moisture content of material is practically unlimited due to the new method of dispersed feeding without increasing the height of the bed. The height of the bed is 500-600 mm as compared to the height of the bed of European apparatus that is more than 1000 mm for the material with high moisture content.

1. THERMAL EFFICIENCY AND CAPACITY

General notion about merits of drying in FB has been based on the increase in the process intensity several times, as compared to all known methods. However, this alone, cannot provide the effective process if, simultaneously, one has not established the possibility to reach high thermal efficiency when using high-temperature heat carrier. The possibility to use gases under initial temperature no less than 600-700°C has a special significance for drying potash salts which coalesce and to form clods under the temperature 0.5 T° of melting point, i.e. at 400-450°C.

Consequently, when using a high-temperature heat carrier one has to eliminate the possibility of salt overheating in the near-the-grid zone. Investigation of the near-the-grid zone structure enabled us to discover a new phenomenon manifesting itself in a sharp and extreme change in structure within the range of high gas velocities (high degree of fluidization). It was found experimentally that the boundary of structure modification in the near-the-grid zone corresponds to lowering the grid surface temperature down to that approaching the temperature of the bed. With gas velocities under this boundary, the grid temperature is close to the heat carrier temperature. Thus, the possibility was found to perform the process with gas temperature exceeding the temperature of forming salt clods on the grid. It should be noted that even relatively small lowering in gas velocity in relation to the boundary value resulted, in practice, in forming clods in the bed and in choking up the grid.

The optimum value of gas velocity may be presented as follows:

$$R_{e\ opt.} = 0.24\ \sqrt{Ar}$$

On the basis of new notions about the peculiarities of hydrodynamic regime, the potash salt drying is carried out at the gas temperature of 650-700°C. In most European units of FB the gas temperature does not exceed 350-400°C. By carrying out the process with high-temperature heat carrier, fuel consumption is cut by 25-35%. Along with that, at the gas velocity corresponding to the bottom boundary of structure modification in the near-the-grid zone, the degree of fluidization greatly exceeds universally conventional parameters. The specific production capacity for potassium chloride drying is growing, therewith, almost twice, thus providing for reduction in grid area, in the process unit overall size, metal consumption and occupied production area. However, performing the process at high specific output reaching 10-15 t of product or 800-1500 kg of moisture evaporated from a square meter per hour requires the development of a new technique for dispersed supply of moist salt into the unit, since under high load one could observe forming of clods at the site of material supply, especially with higher moisture content.

For feed charging, special facilities have been designed which provide dispersed charging (loading) over the bed front by means of special thrower-spreaders. This thrower is located over the bed, on the lateral side of the process unit. These designed thrower-spreaders provide the possibility to supply various, including high-moisture clodforming precipitates.

As production experience had shown the charging of the initial humidity content is, practically, unlimited and may reach 30-70% H_2O for crystallohydrate. We may point out that humid curtain over the bed front during throwing (spreading) allows for reduction of waste gases temperature by 20-30°C.

Carrying out the process at a high degree of fluidization and high initial temperature of gases has allowed for reduction - nearly two times (e.g. potassium chloride drying) - a specific air consumption per unit of evaporated moisture. Accordingly, the electric power consumption for air supply decreases two times.

Thus, the specified technique of the process establishes the possibility to reduce consumption of fuel, electric energy, metal and, accordingly, the cost of equipment, that being the decisive factor for attaining high technical and economic performances.

2. KINETIC CHARACTERISTICS OF THE PROCESS

Practically all the data from literature concerned with drying in fluidized bed evaluate the productivity by taking into account the average time the material stays in the bed. It considers the kinetics has no influence on capacity which is determined only by heat (thermal) balance. In all cases, irrespective of the initial moisture content and the time the material stays in the bed, the moisture content in the material at the process unit outlet is unequivocally determined only by the bed temperature and may be considered as a balanced with relation to the bed temperature.

The importance regarding the independence of drying from kinetics so far has not been recognized by the specialists. Let us dwell on this problem more in detail from the viewpoint of its impact on the optimization of the new production process. The physical model of capacity independence from the time of material staying in the layer and consistency of its moisture content has been made on the basis of a principally new theory by Todes. It treats a fluidized bed as a system where alongside with chaotic particle movement the decisive role is played by the oriented contours of particle circulation under the impact of gravity force from the grid towards the upper boundary of the bed and backwards to the grid. These flows express the ability of the system to self-organizing and specify the value of agitation factor (Dagit.), which depends, according to Todes, on the scale of the system.

As Dagit. specifies the most important property of FB, i.e. its isothermality and isoconcentricity, it becomes comprehensible why the permanence of moisture content at the outlet does not depend on the average time the material stays in the bed, because it is the function of the process unit scale. In the bed the particles get dried many times in the near-the-grid zone, where, as it is seen, takes place the reduction of gas temperature from initial one until the bed temperature and they again moisten near the upper (top) boundary, in the zone of charging. Continuous and multiple rotation of drying and moistening cycles against the background of advantageous agitation provides the consistency of final humidity and its correspondence to a balanced value, even at dewatering of crystallohydrates.

The proposed physical model for drying specifies conditions for optimization of the regime (mode) and choosing a rational process unit design. Inasmuch as the agitation efficiency is an unequivocal function of the process unit scale, it was necessary to specify the parameter of scaling up and its minimum value for reproduction of the process in commercial units.

From our data regarding the relation of the process unit diameter (D) to the bed height (H) (in motionless state) equal to $D/H \geqslant 1$, the parameter for scaling up will be a height of the bed which, regardless of D, equals to $\approx$ 400-500 mm. Agitation efficiency, i.e. Dagit value, if one follows these conditions, will provide the isothermality and isoconcentricity of the process. However, one should take account of the relationship between final humidity and bed temperature which, by our data, is being described by experimentally this relationship :

$U = A1^{-kt}$, where:

U	relative moisture content, kg/kg;
A and K	the constants depending on the physical and chemical nature of the material;
t	bed temperature, °C.

For the salt materials with external moisture content over $\simeq$ 0,8-1%, and where salt is clodded and fluidization is disturbed, the bed temperature should be chosen with regard for this property. When dewatering crystallohydrates we know that the depth of dewatering depends on the consistency between the bed temperature and the temperatures in balanced change of phase composition.

The practical summary taking into consideration the peculiar process kinetics may be stated as follows:

a) an optimum dewatering regime, in general case, represents a one-stage process, with prescribed depth of drying being determined only by the bed temperature;

b) an optimum process unit configuration has to provide a maximum possible efficiency in agitation, therefore, use of rectangle cross-section units having oblong configuration and elongated distance between zone of charging and zone of discharging does not provide a positive effect, but, rather, lowers the agitation efficiency.

c) The search for ways to establish the counter-current regime in the FB dryers contradicts the physical process nature and will not yield a positive result.

During drying, a multiple rotation of drying and particles' dewatering cycles takes place. This is the principal difference of FB from all other methods of conventional drying. On the basis of the concept accepted for optimum organizing of drying we have developed a new pattern for controlling the bed temperature providing for the process stabilization. Inasmuch as the final humidity is the unambiguous function of the layer temperature, it is evident that stability has to be based on maintaining a constant bed temperature with possible variations of moisture content in the material being charged.

As noted above, the drying process is determined by thermal balance, consequently, to control the bed temperature one needs to control a quantity of the moist material being charged, that being achieved by setting in front of the FB unit a surge tank with automatically controlled feeder, while the bed temperature is the pick-up (sensor) for control. This is the pattern implemented at all commercial process units and enables to maintain the bed temperature level, i. e. + 3-5°C.

3. POTASSIUM CHLORIDE DRYING

As noted before, drying potash salts in the FB was implemented in the world for the first time at potash plants in Russia, Western Ukraine and Byelorussia. During the first stages of the commercial process implementation it was decided to compare the operation performances at various plants using the FB units of circular and rectangular cross-section with different types of gas distributing devices. It was established that neither circular units nor those of rectangular cross-section has any significant advantages over each other. The main impact on the stability and efficiency of the process (possibility to increase temperature of the heat carrier) is effected by the design and material of the gas distributing device. The grid must not have elements which hinder particle circulation over its surface, therefore it is not advisable to use cap-like gas distributing devices. There were designs that successfully employed the flat grids with slot and Z-like opening which provide, along with regular fluidization, intensified particle circulation on the grid surface. Discharging of dry product is made precisely at the grid level without threshold, in front of a discharge chute.

In the Ural group of potash plants the FB process units consisted of circular cross-section with Z-formed grids. In Byelorussia and Western Ukraine - the rectangular ones with oblique slots are used. There are dried different grades of potassium chloride: fine, standard flotational and coarse-grained. The initial moisture content is changed from 11-12% (after filtration on filters) down to 6-7% after precipitate centrifuging, and the final moisture content is about 0.05%.

The process conditions:

Gas temperature	750°C
Bed temperature	120-130°C
Bed height (in motionless state)	400-500 mm

The specific air consumption, depending on the material grain size distribution ranges from 4500 to 7000 m^3 per sq.m/hr. The process units have been produced having the grid area from 6,5 to 10 m^2 and with capacity of 80-90 to 130-150 t/hr. The FB units are also used for heating salt prior to its granulating in compactors.

4. DEWATERING OF SCHOENITE ($K_2SO_4 \cdot MgSO_4 \cdot 6H_2O$)

When processing polymineral ore in Western Ukraine one obtains potassium-magnesium fertilizers, i.e. potassium-magnesium sulfate (Kali-magnesia - K_2SO_4 . $MgSO_4$. $6H_2O$) and a number of by-products for production of metallic magnesium in the form of solutions of magnesium chloride and carnallite (KCl . $MgCl_2$. $6H_2O$).

For the first time it was possible to exhaustedly dewater - without recirculation - the schoenite crystallohydrate down to about 0,2% of external moisture content.

In accordance with an optimum bed temperature (170-180°C) the content of crystallization moisture is 3-5% at 28-31% of initial moisture content. In the process of dewatering one gains a coarse product of about 0.8-1,0 mm average particle size and the fines partly dispersed and separated in the cyclones.

Process conditions

Heat carrier temperature	550-600°C
Bed temperature	170-180°C
Bed height	500-550 mm
Specific air consumption	6000 $m^3/m^2/hr$

The process units consist of rectangular cross-section with fan-like slot openings. The units were 8 m^2 grid area at 28-31% of initial moisture content and the capacity is about 20-25 t/hr.

5. DEWATERING OF SALT SOLUTIONS AND CRYSTALLOHYDRATES

The above method is used also for dewatering of various solutions. At the pilot-plant stage tests there was carried out to dewater potassium sulfate and magnesium chloride solutions.

a) **Dewatering of potassium sulfate solutions** - During a number of industrial operations such as synthetic fatty acid (SFA) production from paraffin processing, liquid wastes in the form of salt solutions were produced. In one of the SFA manufacturing options, the wastes produced is in the form of potassium sulfate solution. A finely dispersed product is formed while dewatering potassium sulfate in FB. We have developed a new technique for potassium sulfate granulation while dewatering its solution. This method is based on introducing into solution the compounds promoting formation of granules. Based on our proposed process qualitative classification of ions impact into the process of granulation we have added into the solution a small amount of potassium orthophosphate, and that enables us to obtain the major part of our product in the form of strong and good shaped granules of about 1-2 mm size.

b) **Dewatering the solutions and crystallohydrates of magnesium chloride**

When processing ore from some potash deposits and natural brines there arises the necessity to dewater crystallohydrates and solutions of magnesium chloride. For this group of processes one should take account of thermohydrolysis phenomenon taking place by the following reaction:

$$MgCl_2 + H_2O = MgO + 2HCl$$

The presence of MgO in dewatered material deteriorates its quality, especially when processing it into metallic magnesium. The degree of hydrolysis grows with the temperature of initial moisture content. Therefore the one-stage dewatering cannot be recommended for the group of processes in question.

Among the numerous techniques used for dewatering of magnesium chloride compounds we have developed and tested on the pilot plants and in industrial experiments, a new method developed is as follows: the dewatering of solutions of $MgCl_2$ or carnallite ($KCl \cdot MgCl_2 \cdot 6H_2O$) is made with one step in FB, according to modification developed by us, and it brings down to optimum moisture content in the semi-product. For instance, when dewatering magnesium chloride solutions at the bed temperature of 140-150°C one obtains a mixture of hydrates corresponding to the content of about 2,5 moles of water per one mole of $MgCl_2$. The second stage of dewatering is based on a new technique for processing in FB with an inert particle bed, e.g. corundum. Dewatering takes place on the bed particles' surface and owing to a high intensity of heat exchange the material is heated up to the bed temperature within the contact time period in the order of some seconds and is carried over from the bed into the system of dust suppression. Dewatered product contains moisture corresponding to the bed temperature, but inasmuch as the rate of material in the bed one manages to substantially diminish the MgO content in it.

Dewatering in FB units with inert bed according to the developed method has been used for a number of processes, e.g., when manufacturing ammonium silicon fluoride. Although the bed temperature of 130-140°C exceeds the material decomposition temperature, practically no products of decomposition were found in the waste gases.

The method of dewatering and thermal processing in FB units with inert bed has been tested for quite a few number of materials, e.g.; when processing phosphogypsum into bihydrate, anhydrous gypsum and other products. Its use to replace dewatering is promising in spray dryers, since specific capacity grows by an order of magnitude or more with appropriate decline in metal consumption and production area.

It is advisable to employ the FB units with inert bed for drying finely dispersed materials, e.g., kaolin, gypsum, etc., since the capacity grows therewith many times, as compared to FB units without an inert bed.

In FB units with inert bed processes of calcination at high temperature using the heat of waste gases have been successfully carried out. One can state that this modification of FB will make possible a substantial increase in the process efficiency as compared to calcination in rotary ovens, tube dryers, etc. It is also promising to use this modification for processing of clay slimes from potash operations.

For potassium-magnesium crystallohydrates (kainite ($KClMgSO_4 \cdot 3H_2O$), epsomite ($MgSO_4 \cdot 7H_2O$), etc.) where the dewatering of which is not accompanied by thermohydrolysis, it was shown it was possible to have an efficient one-stage process in which the degree of dewatering is controlled by the bed temperature.