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MATHEMATICAL SIMULATION OF CATALYTIC PASTE FLOW IN A RAM EXTRUDER

A mathematical model was developed for the stage of catalyst paste preparation. The mathematical model allows the calculation of solvate film thickness and volume content of the solid phase at any moment of time. Functional dependencies were found, which correlated the solvate film thickness and volume content of the solid phase with the plastic strength and viscosity of catalyst pastes. A range of plastic strength was determined, which ensures the optimum forming properties of the pastes. The mathematical model allowed the determination of the concentrations of the components of the continuous and dispersed phases, which ensured the optimum paste forming rheological properties, which, in turn, influence the end properties of an α -Fe₂O₃ catalyst. The significant role of the solvate film thickness in the forming properties of pastes was demonstrated.

It is possible to obtain various materials by extrusion: catalysts of different form (from cylinders to figured grains and honeycomb blocks), ceramic materials, food materials (noodles, candies, etc.).

The preparation of materials based on α -Fe₂O₃ is considered in this study. The following stages are involved in the process: synthesis, mixing, extrusion, drying and firing. The obtained materials should have a series of given properties: appearance, durability, porosity, water absorption, etc.

Extrusion is a process in which the agglomeration of paste particles is unwelcome, as it leads to deterioration of the paste properties. Therefore, this paper is devoted to the investigation and mathematical simulation of catalyst paste preparation taking into account the formation of solvate films, which prevent the agglomeration of particles. In this stage, the solid carrier (α -Fe₂O₃) is mixed with aqueous solutions of the surfactants (solutions of poly(vinyl alcohol) – PVA and methyl cellulose – MC). The addition of these substances makes the paste plastic. The substances keep the dispersed phase in a bounded condition, counteracting factors which disintegrate the structure.

The most important problem in the preparation of catalyst pastes is the evaluation and prediction of their forming properties knowing their rheological properties, such as the plastic strength and viscosity [1].

The properties of pastes evolve during the preparation stage. According to physico-chemical mechanics, plastic forming pastes are a system of rigid particles, surrounded by solvate films. The cross-linking of such dispersed systems occurs as a result of the molecular coupling of dispersed phase particles by the

most lyophobic areas of the surface, which are the least protected by the solvate films. The thickness of solvate films significantly determines the technological parameters of the system, including its formability.

EXPERIMENTAL

A series of experiments were carried out in which pastes with different composition were prepared by varying the concentration of the continuous phase (PVA and MC) and the content of the solid phase (Table 1). Each paste was prepared in a mixer for about 30 minutes. Then it matured for 24 hours. The plastic strength was determined after 30 minutes and after 24 hours by means of a Rebinderi conic plastometer [2] by the cone penetration method under the action of constant weight. The viscosity was determined after 24 hours by means of a capillary viscosimeter at a shear strain of 10 s⁻¹.

The experimental values of the plastic strength and viscosity are presented in Table 1.

The behaviour of plastic strength in time was observed for some experiments. The diagram of plastic strength in time has a plateau (Fig. 1), which indicates system stabilization. Only two limiting values of the plastic strength are presented in Table 1: after 30 minutes and after 24 hours. But even these two values are sufficient to prove the importance of taking into account the maturing stage. In 24 hours the plastic strength doubles in almost all the experiments, and in experiment 3 it even quadruples. Structurization of the system occurs during this period, the paste becomes more dense and rigid.

RESULTS AND DISCUSSION

The mathematical model for the stage of catalyst paste mixing was designed on the basis of applying a heterogeneous medium mechanics apparatus and colloid chemistry. This model allows the calculation of

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Table 1. Experimental values of the rheological characteristics of the catalyst pastes

N ^o exp.	α_2^0	C_1^0 kg MC kg paste	C_2^0 kg PVA kg paste	η (when $\dot{\gamma}=10$ s^{-1}), kPa·s	P_m (after 30 minutes), kPa	P_m (after 24 hours), kPa
1	0.502	0.012	0.06	41.20	2059	3658
2	0.460	0.012	0.06	0.78	84	298
3	0.500	0.012	0.018	6.61	566	1304
4	0.458	0.012	0.018	0.36	25	82
5	0.501	0.0012	0.06	15.85	3031	3536
6	0.459	0.0012	0.06	1.12	74	192
7	0.499	0.0012	0.018	0.31	173	318
8	0.457	0.0012	0.018	0.24	32	65
9	0.492	0.0075	0.04	19.01	727	1755
10	0.476	0.0075	0.04	14.79	507	1221
11	0.477	0.00375	0.06	5.75	496	875
12	0.454	0.00375	0.06	0.66	105	180
13	0.484	0.01125	0.02	4.68	289	742
14	0.462	0.01125	0.02	0.56	63	186

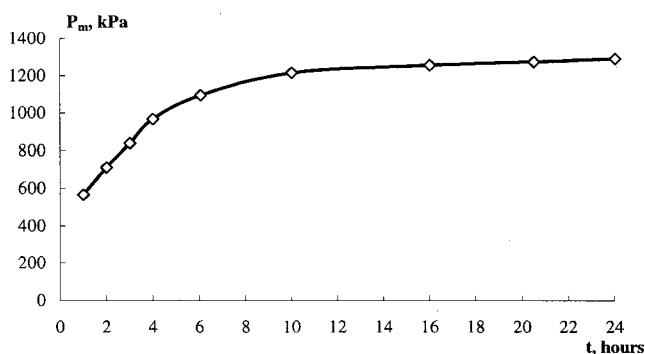


Figure 1. Experimental dependency of the plastic strength on time for experiment 3

the α -Fe₂O₃ particle-size distribution density at any moment in time on solvate film thickness.

A multicomponent two-phase system was considered in the stage of paste preparation in the mixing apparatus. The first phase is continuous, the second one consists of solid α -Fe₂O₃ particles. The components of continuous phase were: water, PVA and MC. We considered that in the mixing process, α -Fe₂O₃ particles were covered by the solvate film. The properties of this film depended on the composition of the continuous phase.

The mathematical model of the catalyst paste mixing stage was constructed on the basis of applying heterogeneous medium mechanics and methods of colloid chemistry. The model allows the calculation of the distribution density of the Fe₂O₃ particles for any moment in time on solvate film thickness and includes the following equations:

– balance equation of the α -Fe₂O₃ particle number, predicting the number of particles at any moment of mixing and storing, having solvate film; predicting the medium size of the solvate film:

$$\frac{\partial f}{\partial t} + \frac{\partial f\mu}{\partial r} = 0; \quad (1)$$

– dependency for the solvate film growth rate:

$$\mu = \frac{dr}{dt} = 4\pi r^2 [K_1 C_1 C_3 + K_2 C_2 C_3^2 + K_3 C_3]; \quad (2)$$

– particle mass change at the expense of the solution components in flux of the continuous phase:

$$\psi = \frac{dm}{dt} = 4\pi r^2 [\rho_{11} K_1 C_1 C_3 + \rho_{12} K_2 C_2 C_3^2 + \rho_{13} K_3 C_3]; \quad (3)$$

– equation of the liquid phase density change:

$$\frac{dp_1}{dt} = \int_0^{R_m} f \psi dr; \quad (4)$$

– equation of the component concentration change for the liquid phase:

$$\rho_1 \frac{dC_i}{dt} = \int_0^{R_m} \rho_{1i} f \mu_i dr + C_i \int_0^{R_m} f \psi dr; \quad (5)$$

where $\mu_1 = 4\pi r^2 K_1 C_1 C_3$, $\mu_2 = 4\pi r^2 K_2 C_2 C_3^2$, $\mu_3 = 4\pi r^2 K_3 C_3$.

The volume content of the solid phase was determined on the basis of these equations (1–5):

$$\alpha_2 = \alpha_2^0 + \int_0^{R_m} r f dr. \quad (6)$$

The unknown kinetic constants of the mathematical model (1–6) are K_1 , K_2 , K_3 . For the definition of the kinetic constants, we took the data represented in the literature as fundamental experimental data [3].

In this paper, the authors introduced the concept of relative thickness of the boundary layers (X/R), which was calculated by means of the porosity coefficient difference (volume of pores to volume of the rigid phase ratio) of the precipitates from the aqueous suspension and a polymer-based dispersion under conditions of free precipitation and after centrifuging for 30 minutes. They obtained a value of this parameter at various polymer concentrations (PVA and MC).

If we take the ratio of two parameters (X/R) at different polymer concentrations, we will see that the radius of the solid phase particles will be cancelled and we will obtain the dependency of the boundary layer magnitude on solid particles at different polymer concentrations. Thus, assuming that the variable h , introduced by us, is analogous to parameter X , we carried out the determination of the kinetic constants. The values of parameter (X/R) at different polymer concentrations are shown in Table 2.

Table 2. Value of the relative thickness of the boundary layers at various polymer concentrations (PVA and MC) [3].

Concentrations, %	MC			PVA			
	0.75	1.5	3.0	0.75	1.5	3.0	6.0
Parameter (X/R)	0.428	0.505	0.581	0.405	0.405	0.421	0.440

The average thickness of the solvate films (h) was calculated at the corresponding polymer concentration. The component of the continuous phase was MC solution in one case and PVA solution in the other. The concentration of MC solutions was 1.5 and 3.0 mass. %, the concentration of PVA was 3.0 and 6.0 mass. %.

The following values of the kinetic constants were determined:

$$K_1 = 10.5 \cdot 10^{-10} \text{ (m}\cdot\text{s}^{-1}), K_2 = 0.66 \cdot 10^{-10} \text{ (m}\cdot\text{s}^{-1}), \\ K_3 = 0.2 \cdot 10^{-10} \text{ (m}\cdot\text{s}^{-1}).$$

The behaviour of such model (1–6) parameters, such as solvate film thickness (Fig. 2) with time is interesting.

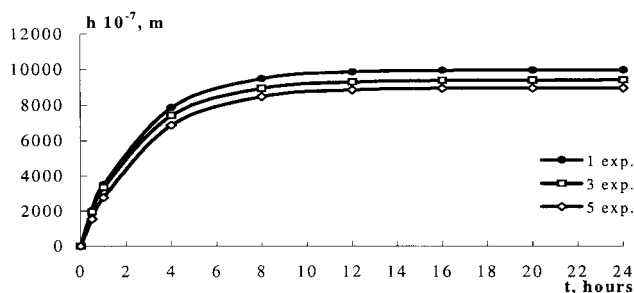


Figure 2. Dependencies of the solvate film thickness on time

In for the first 30 minutes of the mixing stage, growth of the solvate film occurs (Fig. 2). In the stage of storing the growth of solvate films takes place for 5–6 hours and the aging of solvate films takes place for up to 24 hours (the thicknesses tend to a stationary value with time). The stationary values were equal to 8000–10000 Å and depended on the experimental conditions. The higher values of the solvate film average thicknesses are characteristic for the first experiment. This is explained by the higher PVA concentration in the first experiment than in the third one, at equal concentrations of other components in the paste (MC and α -Fe₂O₃) (Tab. 1).

The same type of solvate film thickness change in time was also observed when comparing experiments 1 and 5 (Fig. 2), where the MC concentration changes (Tab. 1). Increase of the MC concentration in the continuous phase leads to significant growth of the solvate film thickness.

The following functional dependencies were obtained by comparing data obtained with the mathematical model (for example, volume content of the

catalyst, taking into account the presence of a solvate film; average values of the solvate film thicknesses) with the experimental data on the plastic and rheological properties of the pastes:

– dependencies for the plastic strength:

$$P_m = 10^3 \exp(-14.792 + 1.77 \cdot 10^6 h + 8.1 \cdot 10^{12} h^2 + \\ + 15.207 \alpha_2 + 61.113 \alpha_2^2 - 2.547 \cdot 10^7 h \alpha_2) \quad (7)$$

– dependencies for the viscosity of the catalyst paste:

$$\eta = \eta_0 \exp(98 \alpha_2 - 36.8) \left(\frac{45.12 \cdot 10^{-7}}{h} - 3.7 \right) \quad (8)$$

Comparison between the data obtained from the dependencies (7, 8) and the experimental data on viscosity and plastic strength is presented in Tab. 3, from which it can be seen that the experimental and calculated data are in good agreement.

The influence of solvate film thicknesses can be seen for all the dependencies. The values of these thicknesses depend on the solid phase components. So, an increase of the solid phase volume content leads to an increase of the plastic strength and viscosity. An increase of the solvate film thickness leads to an increase of the plastic strength and a reduction of the paste viscosity.

Using the obtained dependency (7), we studied the change in plastic strength for 24 hours (Fig. 3). The curve based on the calculated data is in good agreement with the experimental curves. The similar character of the plastic strength change in time indicates that the dependency of plastic strength on the volume

Table 3. Comparison of the calculated and experimental data on plastic strength and viscosity

N ^o exp.	P _m (exp.), kPa	P _m (calc.), kPa	P _m (exp.), kPa	P _m (calc.), kPa	η (exp.), kPa s	η (calc.), kPa s
	After 30 minutes		After 24 hours		After 24 hours	
1	2059	1762	3658	3589	41.20	35.63
2	84	60	298	324	0.78	0.35
3	566	870	1304	1722	6.61	16.38
4	25	52	82	189	0.36	0.29
5	3031	2416	3536	2829	15.85	29.10
6	74	72	192	141	1.12	0.35
7	173	1292	318	1177	0.31	2.44
8	32	63	65	89	0.24	0.27
9	727	637	1755	1230	19.01	11.33
10	507	204	1221	480	14.79	7.10
11	496	228	875	458	5.75	2.23
12	105	47	180	120	0.66	0.19
13	289	346	742	810	4.68	4.92
14	63	70	186	239	0.56	0.45

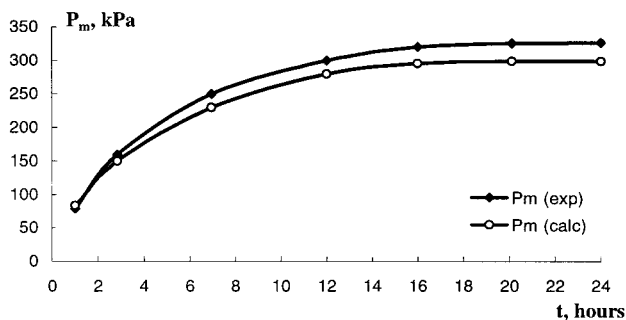


Figure 3. Comparison of the experimental and calculated curves of the change in plastic strength with time for experiment 2

content of the solid phase and solvate film thickness, that we determined, adequately describes the system behaviour in time.

The time of system stabilization coincides with the experimental one (outcome to plateau in Fig. 3) and is 16 hours for the second experiment.

We developed a mathematical model of catalyst paste flow for a RAM extruder. The total pressure can be presented as the sum of two components [4]:

1. Pressure in the compression zone P_1 :

$$P_1 = (\sigma_0 + \alpha V^n) \ln \left(\frac{S_0}{S} \right) \quad (9)$$

2. Pressure in the die P_2 :

$$P_2 = \frac{2kL}{R} \left[(V - V_{slide}) \frac{3n+1}{nR} \right]^n, \quad (10)$$

where $V_{slide} = aV$ ($a < 1$), $\tau = k\gamma^n$.

Then the total pressure:

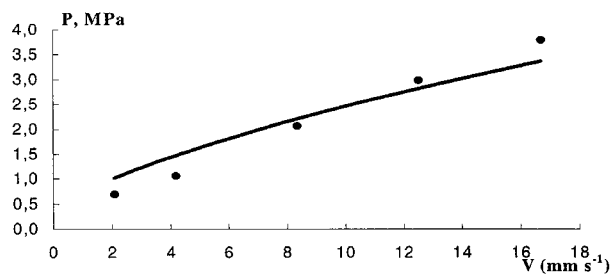
$$P = (\sigma_0 + \alpha V^n) \ln \left(\frac{S_0}{S} \right) + \frac{2kL}{R} \left[V(1-a) \frac{3n+1}{nR} \right]^n. \quad (11)$$

The unknown parameters in the mathematical model are the following parameters: σ_0 , α , k , n and V_{slide} (or a). Parameters σ_0 , α , k , n are constants, which characterize the paste and do not depend on the extrusion conditions. Parameter a (ratio of the paste slide velocity near the die wall to the average paste velocity) is a function of the die radius. It decreases when the radius increases, since the friction of the surface of the catalyst paste in the die wall increases.

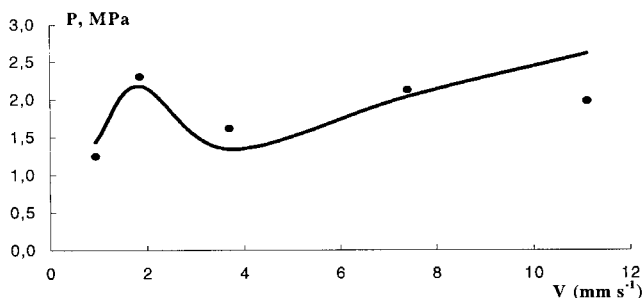
The selection of these parameters was carried out by the method of random search. The unknown parameters were selected so that the divergence between the experimental and calculated data was minimal simultaneously for three dies with which the experiment was realized.

As an outcome of the model parameter selection, the following results were obtained:

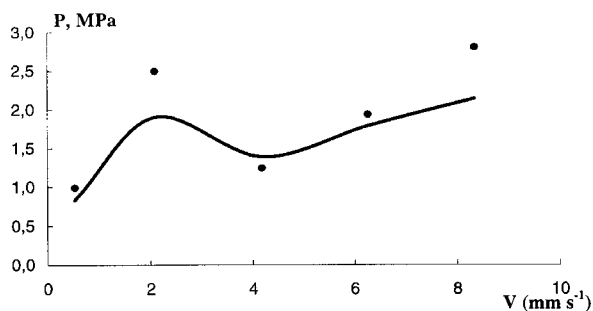
$$n = 0.68, \sigma_0 = 80 \text{ kPa}, \alpha = 40 \text{ Pa} \cdot (\text{mm s}^{-1})^n, \\ k = 1.8 \text{ (kPa s}^n).$$



a



b



c

Figure 4. Comparison of the experimental (points) and calculated (curve) data of the pressure values P upon paste flow velocity for capillaries with different diameters: a) $D = 2 \text{ mm}$, $L = 16 \text{ mm}$, $a = 0,9$; b) $D = 3 \text{ mm}$, $L = 24 \text{ mm}$; $a = 0$ at $V < 4 \text{ (mm s}^{-1})$; $a = 0,82$ at $V > 4 \text{ (mm s}^{-1})$; c) $D = 4 \text{ mm}$, $L = 32 \text{ mm}$, $a = 0$ at $V < 4 \text{ (mm s}^{-1})$; $a = 0,74$ at $V > 4 \text{ (mm s}^{-1})$.

Comparison of the calculated values of the pressure with the experimental data is represented in the graphs (Fig. 4).

A dependency between the consistence constant k (h , α_2), the average thickness of the solvate film (h) and the volume content of the solid phase (α_2) was found. By substituting the last one into equation (11), one can obtain the equation for the relation between the extrusion pressure and solvate film thickness, the volume content of solid phase or, in other words, the initial characteristics of the paste (concentrations of the components in the solid and continuous phase):

$$P = (\sigma_0 + \alpha V^n) \ln \left(\frac{S_0}{S} \right) + \\ + \frac{2 \left[\exp(98\alpha_2 - 36.8) \left(\frac{0.129}{h} - 0.01 \right) \right] L}{R} \left[V(1-a) \frac{3n+1}{nR} \right]^n \quad (12)$$

A dependency was obtained, which correlated the property of the end product (mechanical durability) with the initial data (α_2^0) for the preparation of catalyst pastes and the rheological properties (P_m):

$$\omega = -4.69 + 11.923\alpha_2^0 + 3.196 \cdot 10^{-5} P_m \quad (13)$$

The optimal paste, in our opinion, is the one with a composition, ensuring high plastic rigidity and low viscosity. Low viscosity ensures a decrease of energy usage for extrusion. The high rigidity of the paste, at other equal conditions, will ensure its value in the end products (extrudate), good enough to ensure end product utilization. It was shown experimentally, that these conditions are met by the pastes with plastic rigidity equal to 500 ÷ 1200 kPa, and viscosity – 0.5 ÷ 1.5 kPa·s. Outside of these ranges, the possibility is high that the paste will not flow through the die. It can be seen from equation (8), that the minimum value of the viscosity was obtained at the maximal value of the solvate film thickness and the minimal value of the solid phase volume content. The same rule is true for the determination of the minimal extrusion pressure (12). Therefore, the concentrations of the solid and continuous phase components were varied in a way to have the maximal value of plastic rigidity (in the experimentally found interval), but at the same time to have a paste viscosity in the upper stated range.

The result of recipe selection is shown as a table, where different recipes are shown as well as the corresponding calculated values of the rheological properties (viscosity, plastic rigidity), deformation properties (elasticity) and properties of the end products (porosity, mechanical strength). Using this table, one can select the paste composition that meets the required conditions.

CONCLUSIONS

A mathematical model was developed for the stage of catalyst paste preparation, allowing the calculation of the solvate film thickness of the solid phase and the solid phase volume content at any moment in time.

Functional dependencies were found for the plastic rigidity and viscosity as a function of the solvate film thickness and solid phase volume content. Increase of the solid phase volume content led to an increase of the plastic rigidity and viscosity. Increase of the solvate film thickness led to an increase of the plastic strength and a decrease of the paste viscosity. Comparison between the calculated and experimental data showed the possibility of applying these dependencies to predict the possible values of the plastic rigidity and viscosity of the catalyst paste.

A mathematical model was developed for paste flow in an extruder. It was shown that the obtained dependency between the extrusion pressure and paste

extrusion rate could be applied for dies with any number of channels with known diameter.

It was found that the rate of side shear depended on the capillary diameter and decreased when the channel diameter increased. It was shown that at low rates (lower than 2 mm/s under the conditions of the performed experiment) shear was absent, which could be explained by the existence of some critical rate, the exceeding of which led to occurrence of the shear effect.

A table of paste recipe selection was obtained, which could help in the selection of paste composition, which would provide the required conditions on the rheological, deformation properties and properties of the end product.

NOMENCLATURE

- a – Ratio of the paste slide velocity near the die wall to the average paste velocity;
- C_1 – Concentration of MC in the continuous phase, $\frac{\text{kg MC}}{\text{kg paste}}$;
- C_2 – Concentration of PVA in the continuous phase, $\frac{\text{kg PVA}}{\text{kg paste}}$;
- C_3 – Concentration of free water in the continuous phase, $\frac{\text{kg H}_2\text{O}}{\text{kg paste}}$;
- C_1^0 – Initial concentration of MC in the continuous phase, $\frac{\text{kg MC}}{\text{kg paste}}$;
- C_2^0 – Initial concentration of PVA in the continuous phase at to, $\frac{\text{kg PVA}}{\text{kg paste}}$;
- R – Radius of the die, m;
- f – Distribution function of the $\alpha\text{-Fe}_2\text{O}_3$ particle number with film thicknesses from r to r + dr, $\text{m}^3 \text{m}^{-3}$;
- h – Solvate film thickness, m; k – Constant, degree of consistence, Pa s^n ;
- K_i – Kinetic constants of the i-th component in the mathematical model, m s^{-1} ;
- L – Length of the die, m;
- l – Average diameter of the $\alpha\text{-Fe}_2\text{O}_3$ particles, m;
- n – Viscosity parameter;
- P – Total pressure in the ram extruder, Pa;
- P_1 – Pressure in the compressions zone, Pa;
- P_2 – Pressure in the die, Pa;
- P_m – Plastic strength, Pa;
- R_m – Solvate film maximum volume, m^3 ;
- S – Cross-sectional area of the die, m^2 ;
- S_0 – Cross-sectional area of flow, m^2 ;
- t – Time, s;
- V – Mean extrudate velocity in the die, m s^{-1} ;
- V_{slide} – Sliding velocity, m s^{-1} ;
- X – Length of the boundary layer, m;
- α – Factor of velocity development, $\text{Pa} \cdot (\text{mm s}^{-1})^n$;
- α_2^0 – Initial volume content of the solid phase;
- α_2 – Volume content of the solid phase;
- γ – Rate of shear strain, s^{-1} ;
- η – Viscosity of the catalyst paste, Pa s;
- η_0 – Viscosity of the continuous phase, Pa s;
- μ – Solvate film growth rate, $\text{m}^3 \text{s}^{-1}$;

- μ_1 – Solvate film growth rate at the expense of MC adsorption, $\text{m}^3 \text{s}^{-1}$;
 μ_2 – Solvate film growth rate at the expense of PVA adsorption, $\text{m}^3 \text{s}^{-1}$;
 μ_3 – Solvate film growth rate at the expense of free water adsorption, $\text{m}^3 \text{s}^{-1}$;
 ρ_{11} – Density of the MC solution in the continuous phase, kg m^{-3} ;
 ρ_{12} – Density of the PVA solution in the continuous phase, kg m^{-3} ;
 ρ_{13} – Density of water, kg m^{-3} ;
 ρ_1 – Density of the continuous phase, kg m^{-3} ;
 σ_0 – Shear stress at the entrance of the die, Pa;
 τ – Shear stress, Pa;
 ω – Mechanical strength of the catalyst pastes, Pa;
 ψ – $\alpha\text{-Fe}_2\text{O}_3$ particles mass change, kg s^{-1} .

Indexes:

- 1 – Methyl cellulose (MC);
 2 – Poly(vinyl alcohol) (PVA);
 3 – Water (H_2O).

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IZVOD**MATEMATIČKA SIMULACIJA STRUJANJA KATALITIČKE PASTE $\alpha\text{-Fe}_2\text{O}_3$ U RAM EKSTRUDERU**

(Naučni rad)

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U radu je razvijen matematički model kojim se može opisati jedna faza u pripremi paste od $\alpha\text{-Fe}_2\text{O}_3$ koja ima određena katalitička svojstva. Model omogućava izračunavanje debljine filma rastvarača i zapreminskog udela čvrste faze-paste ($\alpha\text{-Fe}_2\text{O}_3$) u funkciji vremena. Utvrđene su odgovarajuće funkcionalne zavisnosti koje povezuju debljinu filma rastvarača i zapreminskog udela čvrste faze ($\alpha\text{-Fe}_2\text{O}_3$) sa silom plastične deformacije i viskozitetom paste (katalizatora). Utvrđena je oblast vrednosti smicajnih napona pri kojima dolazi do plastičnih deformacija paste, a koje obezbeđuju optimalne uslove formiranja poželjnih osobina koje treba da ima pasta u procesu formiranja ekstrudata $\alpha\text{-Fe}_2\text{O}_3$.

Matematički model omogućava izračunavanje koncentracija kontinualne i dispergovane faze, pri kojima se ostvaruju optimalne reološke osobine paste pri njenom formiranju, koje na kraju imaju uticaja na krajnje karakteristike $\alpha\text{-Fe}_2\text{O}_3$ katalizatora. Pokazano je u kojoj meri je značajna debljina filma rastvarača u formiranju odgovarajućih osobina paste odnosno katalizatora.

Key words: Iron oxide • Elastic properties • Catalysis • Growth mechanism.

Ključne reči: Feri oksid • Elastične osobine • Kataliza • Mehanizam rasta •