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Effect of Microwave Radiation on the Development of a Porous Aluminum Carrier of the Active Mass of Catalysts in Heterogeneous Catalytic Reactions

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Abstract

In this study, we prepared a fine-grained, aluminum-reinforced alumina support matrix of the active mass of catalysts, which showed activity in heterogeneous catalytic reactions stimulated by electromagnetic radiation in the microwave range. The support absorbed microwave radiation of 2.45 GHz. We compared the textural characteristics of samples of Al/Al_2O_3 supports obtained by conventional heat treatment to those synthesized by heat treatment in a microwave field. We found several advantages of heat treating potential supports of the active mass by using microwave radiation. Using the examples of joint deep oxidation of n-butane and carbon monoxide, dealkylation of toluene with water vapor into benzene, and acidylation of diethyl amine with m-toluic acid, we found that after exposure to microwave radiation, the fine-grained aluminum powder-reinforced Al/Al_2O_3 support can be used for preparing catalysts that can be used in reactions stimulated by a microwave electromagnetic field.

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Keywords

Microwave radiation; heat treatment; heterogeneous catalysis; active mass support; reinforcement; fine-grained aluminum; gamma-alumina; absorption of microwave field energy; thermos-transformation properties; radiation penetration depth; textural properties

1. Introduction

New methods based on the absorption of energy from a microwave electromagnetic field for the heat treatment of materials have been developed in various fields of research, such as those associated with the preparation of catalysts and the stimulation of catalytic reactions [1, 2].

The replacement of heating media with microwave energy in the laboratory or the industry has greatly simplified catalyst preparation and improved operating efficiency. As the level of atmospheric emissions associated with heating decreases, chemical production technology becomes more environmentally friendly [3, 4].

The course of heterogeneous catalytic reactions in a microwave electromagnetic field is largely determined by the heat that is released during the absorption and transformation of microwave energy by the catalysts used and is sufficient for the process to advance [5, 6]. The maximum transformation of microwave energy possible in the reaction medium influences the consumption of energy during the catalytic process in a microwave field. An important characteristic of solid-phase catalysts in microwave radiation-stimulated processes is the depth of penetration of the electromagnetic wave into the catalytic charge.

Heterogeneous catalysts of the supported type mainly consist of inert support matrices (aluminum oxide, silica gels, and zeolites with different modifications), which, as dielectrics, poorly absorb microwave radiation. Thus, it is necessary to improve this characteristic and convert the absorbed radiation into heat.

In our previous studies [7-9], we found that industrial-grade silica gel has the lowest microwave absorption capacity among the common industrial carriers (such as KSM, ShSM, KSK, ShSK (GOST 3956–76), A-1, A-64 (GOST 23201.1–78) aluminum oxide, natural zeolites (clinoptilolite and mordenite from Aydag and Chandana deposits of Azerbaijan) [10], and samples of γ-Al_2O_3 prepared under laboratory conditions). Although industrial and lab-made samples of aluminum oxide are better than silica gel in absorbing radiation, they are still unable to convert this radiation into heat required to initiate a catalytic conversion.

Structural composites are quite promising for conducting reactions in a microwave electromagnetic field. In structural composites, finely dispersed metal ferromagnetic, ferrimagnetic, or other electrically conductive particles with an amorphous or crystalline structure are distributed across a porous dielectric matrix. Such materials act as an inert matrix for supported catalysts with high dielectric losses [11-13].

In this study, we used a universal microwave-absorbing alumina support matrix for the active mass of catalysts that showed activity in heterogeneous catalytic reactions stimulated by electromagnetic radiation in the microwave range.

2. Experiments

The alumina matrix was reinforced with fine particles of metallic aluminum by dispersing a specific amount of aluminum powder in a freshly deposited aluminum hydroxide hydrogel (1.0–3.0 wt%), followed by structuring the compositions under the conditions of hydrothermal synthesis and exposure to microwave radiation.

The compositions of PA-1 and PA-2 industrial grade aluminum powders (with grain size distributions of 45.0–63.0 μm and 25.0–45.0 μm, respectively, as defined in GOST 6058–73) underwent hydrothermal treatment in the presence of the freshly deposited aluminum hydroxide hydrogel. The treatment was conducted in a sealed electric coil-heated autoclave made of grade 12X18H10T stainless steel and designed to withstand a positive pressure of 5 MPa. It was equipped with a reference pressure gauge and a shut-off system at the vapor inlet/outlet.

The samples were treated with water vapor and heated at 473–523 K for 5–10 h. They were removed from the autoclave and subjected to heat treatment in a microwave electromagnetic field.

The heat treatment was performed using an installation equipped with an EM-G5593V microwave oven (Panasonic) with a 23 L capacity resonator. The maximum input of the magnetron was 200–900 W, and the operating frequency was 2,450 MHz.

The microwave oven performance facilitated regular heat treatment of the samples with an electric coil and also the adjustment of the microwave radiation power to electrical heating ratio using a software program. The temperature of the samples in the oven resonator was measured using a VA6520 infrared pyrometer with a scale range of -50°C to +600°C.

The loss of microwave radiation power in the mass of the catalyst samples (P_X) and the depth of radiation penetration into the catalytic charge (δ_E) were assumed to be the criteria for evaluating the efficiency of the thermo-transformation properties [14, 15].

The loss of radiation power that occurred while passing through the catalytic charge was determined based on the equivalent conversion of the microwave field energy into heat and an increase in the temperature of the calorimetric body (distilled water) that absorbed the energy:

$$\boldsymbol{P_x = \frac{c \cdot m \cdot \Delta T}{0,24\tau} = \frac{4,17 \cdot m \cdot \Delta T}{\tau}} \ \text{(J/s)}, \qquad (1)$$

Here, 0.24 is the heat equivalent of work; m denotes the mass of water (g); c denotes the specific heat capacity of water = 4.187 kJ/kg*K; ΔT denotes an increase in the temperature of water (K); τ denotes the exposure time in the microwave oven (s).

To determine the depth of electromagnetic wave penetration into a catalytic charge of a complex composition, the thickness of the catalyst layer ($\boldsymbol{\delta_E}$) needs to be determined to ensure almost complete absorption of microwave energy by the object exposed to it:

$$\boldsymbol{\delta_{\mathrm{E}} \approx \frac{\lambda}{\pi\sqrt{2\varepsilon'(\sqrt{1+tg^2\delta}-1)}}} \ \text{(mm)}, \qquad (2)$$

Here, δ_E denotes the distance at which the amplitude of the electric field strength vector E_0 decreases by a factor of e (e ≈ 2.7, the base of the natural logarithm), ε' denotes the real part of the

relative permittivity of the catalyst material, tgδ denotes the tangent of the dielectric loss angle, and λ denotes the wavelength.

The textural characteristics of the synthesized Al/γ-Al_2O_3 samples were studied using a unified Sorbi-MS installation and calculated using the BET method (flow measurement of nitrogen adsorption isotherms at 77 K).

The X-ray phase analysis and the assessment of the microstructure of the powdered samples were performed using a DRON-3 diffractometer in filtered radiation of CuKα range (nickel filter) at 30 kV and 20 mA in the X-ray tube. The resulting diffractograms were analyzed using the JCPDS database. The average size of the crystallites of the γ-Al_2O_3 phase (areas of coherent dispersion) was estimated from the broadening of reflections in diffractograms using the Debye-Scherrer formula [16]:

$$\boldsymbol{D = \frac{k \cdot \lambda}{\beta \cdot Cos\theta}} \quad (3)$$

Here, **D** denotes the average crystal size, **λ** denotes the wavelength of the X-ray tube, **β** denotes the diffraction peak width at half the height, **θ** denotes the diffraction angle, and **k** = 0.9 (correction factor).

The dimensions of the sample microcrystallites were determined and compared based on the results obtained using a JSM-6460LV high-resolution SEM with a 7,500 F field emitter (JEOL, Japan). The images were fixed in a low-energy SE mode.

3. Results and Discussion

During hydrothermal synthesis, metastable forms of aluminum oxide (pseudoboehmite and boehmite) were formed on the surface of aluminum crystallites based on a common reaction [17].

$$\boldsymbol{2Al^0 + 2H_2O + O_2 \rightarrow 2AlOOH + H_2} \quad (4)$$

The reaction promoted a good combination of unreacted aluminum microcrystallites with aluminum hydroxide hydrogel introduced into the reaction zone.

In the confined space of the autoclave, an increase in the volume of the reaction charge resulted in its consolidation and led to the formation of large quantities of samples containing an aluminum-reinforced aluminum-hydroxide phase.

The amount of unreacted (4) aluminum in the samples was determined by the method described in another study [18] using the volumetric method and based on the volume of hydrogen that was released when the test portion of the sample was treated with a 30% solution of NaOH.

The relationship of the degree of conversion of samples of aluminum powders of various dispersed compositions with the time and temperature of hydrothermal treatment of the composition (Al + aluminum hydroxide hydrogel) is shown in Figure 1.

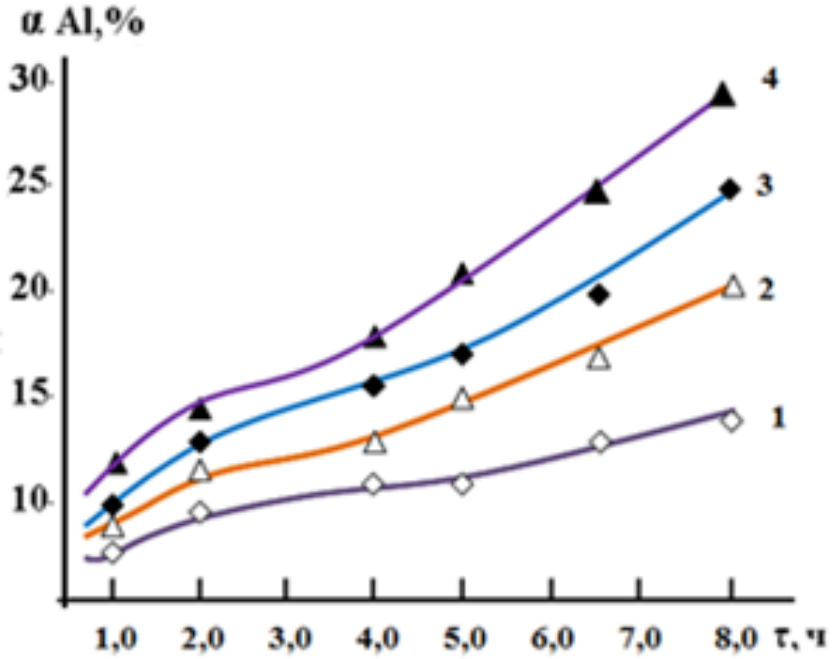

Figure 1 The relationship between the conversion degree of aluminum powders PA-1 (1.2) and PA-2 (3.4) and the hydrothermal synthesis time. Reaction temperature (1.3) - 473 K, (2.4) - 523 K. The initial content of Al-powders in the composition was 3.0 wt%.

Under comparable reaction conditions (4), powder samples with smaller grain sizes (PA-2) showed higher reactivity, and thus, the required degree of conversion was completed over a shorter synthesis time.

Based on these outcomes, it is expected that for reinforcing metallic aluminum powders (PA-2), the hydrothermal treatment conditions might be adjusted to obtain a material that brings together a dielectric matrix (as a nonconductor) and current-conducting elements with linear sizes close to those of the "skin layer" [19] and absorbs microwave radiation instead of reflecting it.

Based on the relationship between the variable power of the magnetron and the temperature trends of $Al/Al(OH)_3 \cdot nH_2O$ compositions reinforced by Al-powders of different grain size distributions and the freshly deposited aluminum hydroxide hydrogel, we concluded (Figure 2) that all initial samples, which contained free-water, absorbed microwave radiation at a relatively low output value because of high dielectric losses ($tg\delta\ H_2O = 0.11$ at 20°C).

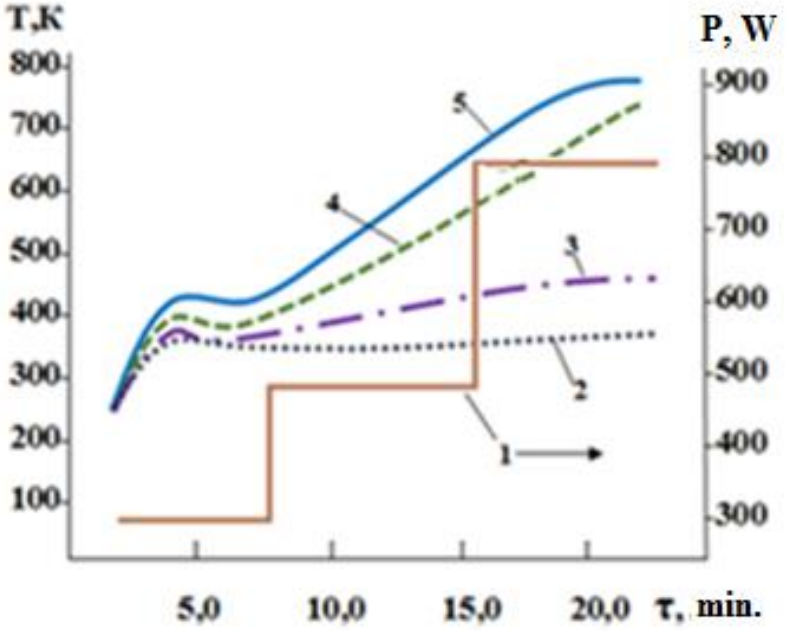

Figure 2 Variation of magnetron power - (1) and temperature change of the freshly deposited hydrogel ($Al(OH)_3 \cdot nH_2O$) - (2); the composition of $Al(OH)_3 \cdot nH_2O$ with PA-1 powder (α = 30%) - (3), PA-1 powder (α = 10%) - (4), and PA-2 powder (α = 10%) - (5).

Based on equation (5) (modified), the sample temperature shift reached an extreme value and stabilized at around 373–383 K (until the complete evaporation of water) due to the balance of absorption and heat loss.

$$\Delta \boldsymbol{T} = \frac{\boldsymbol{P_{def.}} \cdot \boldsymbol{\tau}}{\mathrm{c} \cdot \boldsymbol{d}} \ (K), \qquad (5)$$

Here, P_{def} denotes the microwave radiation power absorbed by a unit volume of $Al/Al(OH)_3 \cdot nH_2O$ (W/m^3); τ denotes the time of field exposure (s); c denotes the average heat capacity of the tested samples (kcal/deg*mol); d denotes the density of the samples (g/cm^3).

The samples that did not contain inclusions of metallic aluminum and those with a relatively low residual content after hydrothermal treatment (~1 wt% at α ≥ 30%) showed no or negligible levels of microwave energy absorption even at the maximum emission output due to the small value of the dielectric loss tangent ($tg\delta = 1 \cdot 10^{-4}$ for Al_2O_3). The presence of 1.5–2.0 wt% aluminum crystallites in the initial composition promoted an increase in dielectric losses of microwave radiation and its transformation into heat, provided that the conversion of aluminum in the hydrothermal process brought this value to ~10%. The process was accompanied by an increase in the bulk temperature of the samples, which reached 663–773 K at a magnetron power of 800 W and a heating rate of ~20–25 nK/min. This caused bound water to be removed from the exposed samples, and a gamma-form alumina matrix was formed.

The X-ray diffraction patterns of the γ-Al_2O_3 sample obtained through conventional heat treatment and the Al/Al_2O_3 matrix sample synthesized through heat treatment in the microwave field are shown in Figure 3.

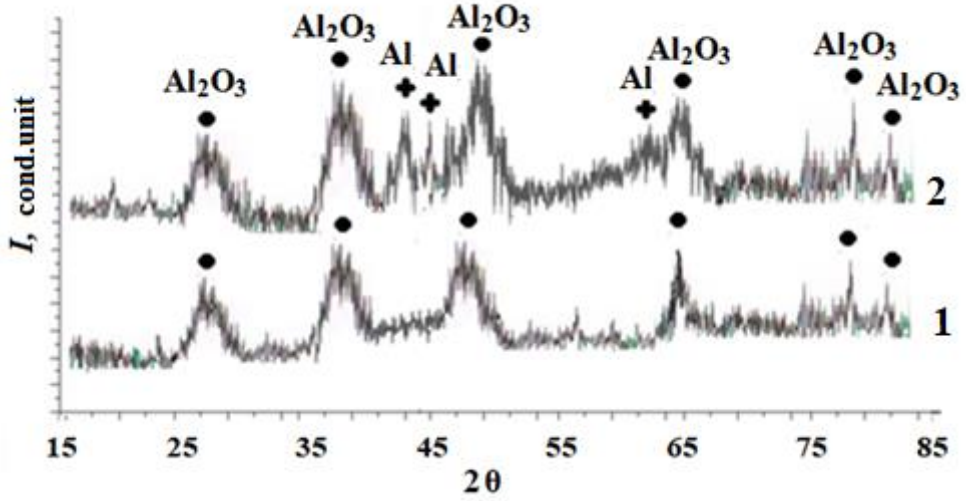

Figure 3 The X-ray diffraction patterns of the γ-Al_2O_3 sample (1) and microcrystalline-reinforced Al γ-Al_2O_3 matrix (2). **+** - Al, ● - γ-Al_2O_3.

Based on the similarities of the reflections of the γ-Al_2O_3 phase on the diffractograms of both samples, we concluded that a gamma-alumina phase formed through the microwave synthesis of the Al/Al_2O_3 matrix was identical to that formed by conventional heating. The spectrum (2) showed reflections from inclusions of aluminum microcrystallites in the alumina matrix.

As the average dispersion degree of the aluminum powders increased (PA-1 < (PA-1 + PA-2) < PA-2), a line with increasing intensity (d/n = 1.95 Å) appeared in the diffractograms of the Al/Al_2O_3 samples obtained from such powders along with reflections from the γ-Al_2O_3 phase. This line indicated highly crystallized boehmite [20]. However, the line did not appear in the spectrum of the composite sample obtained using coarse PA-1 powder (Table 1).

Table 1 The XRD data: Alumina samples reinforced with PA-1 and PA-2 industrial grade aluminum powders were obtained by heat treatment in a microwave field. Conditions: magnetron input = 800 W and exposure time = 15 min.

Al/Al_2O_3 composition (PA-1 powder, 1.0 wt%)		**Al/Al_2O_3 Powder mix: PA-1 (0.5 wt%) and PA-2 (0.5 wt%)**		**Al/Al_2O_3 composition (PA-2 powder, 1.0 wt%)**	
d/n, Å	**I**	**d/n, Å**	**I**	**d/n, Å**	**I**
4.54	3	4.49	7	4.46	7
2.76	54	2.77	52	2.81	52
2.42	56	2.40	53	2.36	53
2.30	28	2.28	27	2.27	27
1.98	74	1.99	55	1.99	55
-	-	**1.95**	19	**1.95**	31
1.52	25	1.52	35	1.53	35
1.40	100	1.40	100	1.41	100

These results indicated the advantage associated with the pseudoboehmite - boehmite - gamma-alumina phase transitions during the formation of the support matrix with the introduction of a finer-grained PA-2 powder.

The heat treatment method used for the synthesis of the Al/γ-Al_2O_3 support samples influenced their surface morphology.

The images of the Al/γ-Al_2O_3 support samples obtained by conventional heat treatment and microwave exposure are shown in Figure 4 and Figure 5.

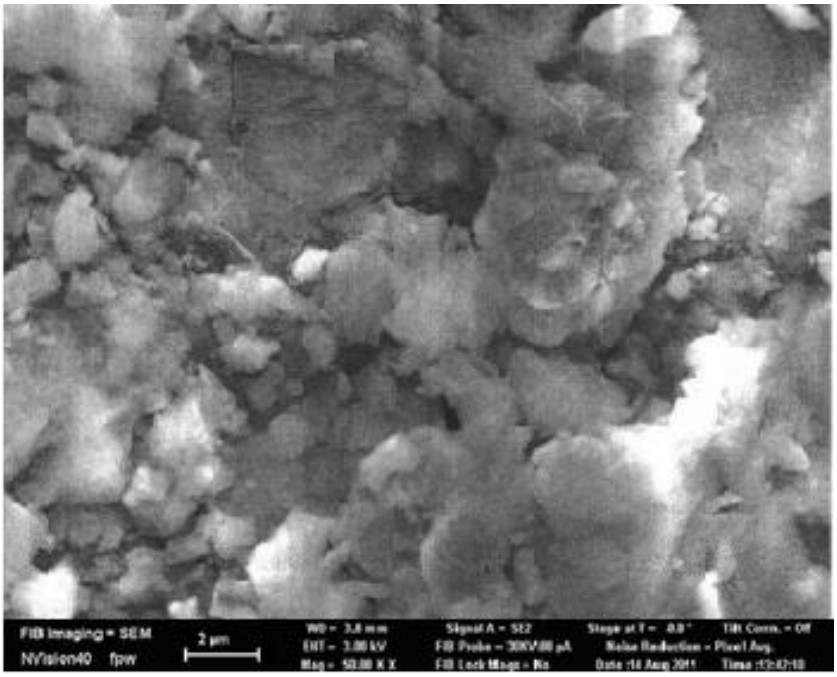

Figure 4 An image of a γ-Al_2O_3/Al support sample obtained by conventional heat treatment.

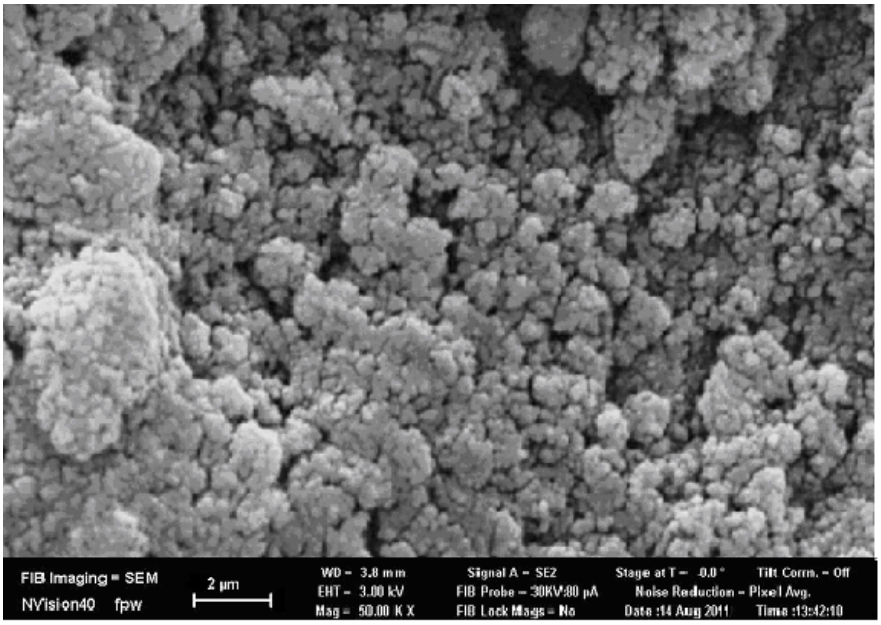

Figure 5 An image of a γ-Al_2O_3/Al support sample obtained by heat treatment in a microwave field.

The effective pore radius was also redistributed in the process, and pores of medium and large size were formed.

The heat treatment of the samples in the microwave field resulted in a finer-grain surface structure. The surface texture became more prominent, which ultimately increased the specific surface area and porosity (Table 2).

Table 2 The textural characteristics of composite Al/Al_2O_3 support reinforced with aluminum powders of various dispersed compositions after heat treatment in a microwave field.

Support sample	Heat treatment conditions in microwave field (T (K)/exposure time)		$S_{def.}$ (m^2/g)	V_{pore} (cm^3/g)	Distribution of pore space by size (cm^3/g)		
	Drying	Calcination			Pore diameter (nm)		
					5–10	10–100	100–1000
Al/Al_2O_3 (PA-1)	378/10 min	723/12 min	182	0.58	0.30	0.22	0.06
		753/14 min	175	0.57	0.26	0.23	0.08
Al/Al_2O_3 PA-1 + PA-2	378/10 min	723/12 min	167	0.56	0.28	0.18	0.10
		753/14 min	165	0.56	0.25	0.19	0.12
Al/Al_2O_3 (PA-2)	378/10 min	723/12 min	158	0.53	0.26	0.16	0.11
		753/14 min	155	0.50	0.23	0.13	0.14
Pseudobohemit*	383/2 h	773/4 h	235	0.59	0.41	0.12	0.06
Bohemit*	383/2 h	773/4 h	98	0.43	0.10	0.15	0.18

* The samples were heat treated by conventional heating.

The textural characteristics of Al/Al_2O_3 support samples reinforced with PA-1 and PA-2 powders that were formed by subsequent heat treatment in a microwave field are presented in Table 2. To determine the efficiency of microwave synthesis, the properties of gamma-alumina samples obtained by traditional electric heat treatment are also presented.

The heat treatment in a microwave field increased the rate of formation of the porous structure of Al/Al_2O_3 supports compared to the rate achieved for alumina matrices subjected to conventional heating. Additionally, as the dispersion of the reinforcing aluminum powders increased with the temperature and exposure time in the microwave field, the specific surface area and the pore space of the Al/Al_2O_3 composite samples decreased.

The observed regularities were consistent with our assumption regarding the effect of possible phase transitions (pseudoboehmite-boehmite-gamma alumina) in the initial stages of preparation of aluminum-reinforced alumina supports on their porous structure, which was formed by heat treatment in the microwave field.

By varying the content of aluminum microcrystallites introduced into the alumina matrix, an extremal relationship between microwave power loss in the material of the synthesized γ-Al_2O_3/Al composition and the concentration of the reinforcing particles was established, with a maximum

content of aluminum within 1 wt% (Figure 5). In this case, the average size of the γ-Al_2O_3/Al crystallites was 120 nm.

For a similar content of the reinforcing additive, the penetration depth of microwave radiation showed the lowest value at a frequency of 2.45 GHz.

Based on the data presented in Figure 6, we concluded that the increase in the microwave power loss during the interaction with the reinforced γ-Al_2O_3 support, which was in the range of the optimum values of the filler concentration, was associated with the effective absorption of radiation energy by metal particles whose size was comparable to that of the skin layer.

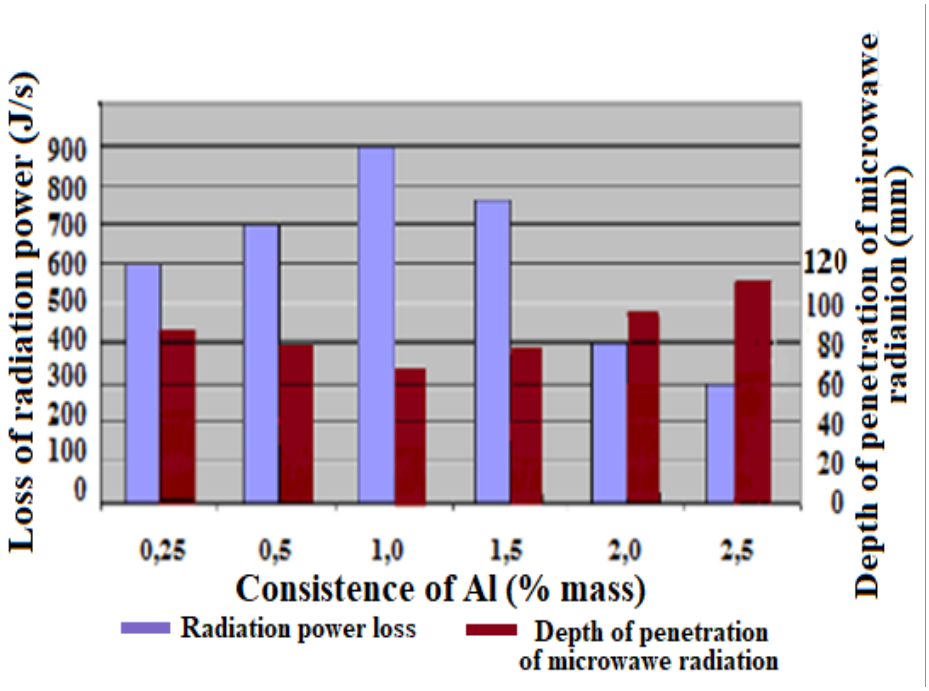

Figure 6 The relationship of thermo-transformation properties of the γ-Al_2O_3/Al composition with the depth of microwave radiation penetration and the volumetric concentration of reinforcing particles. Conditions: magnetron power = 800 W, exposure time = 2.5 min.

By varying the hydrothermal treatment conditions, we could change the degree of conversion of aluminum microcrystallites according to the course of the reaction (4), which in turn allowed us to adjust their dimensions.

Thus, by performing hydrothermal treatment of industrial aluminum powders in the presence of the aluminum hydroxide hydrogel followed by sintering in a microwave field, we obtained a γ-Al_2O_3/Al support of the active mass of catalysts for microwave radiation-stimulated reactions, which intensively absorbed the energy in a microwave field (2.45 GHz frequency).

The average heating temperature of γ-Al_2O_3/Al support samples containing the microcrystalline PA-2 aluminum powder as a reinforcing material and the heating rate achieved by the action of microwave radiation of varying intensities were derived from the data in Figure 2. They were comparable to the temperature required for the known redox and acid-base heterogeneous catalytic reactions [21-23] with conventional heating of the catalytic charge.

If the active catalyst components deposited on the surface of the γ-Al_2O_3/Al support are also microwave radiation-absorbing materials, the thermal transformation effect of such catalysts should increase [24, 25].

This regularity was confirmed by the results provided in Table 3 and was based on the parameters of joint deep oxidation of n-butane, and carbon monoxide, dealkylation of toluene with water vapor into benzene, and acidylation of diethyl amine with m-toluic acid; all reactions were performed under optimum conditions with either conventional or microwave heating.

Table 3 The reaction parameters of joint deep oxidation of n-butane and carbon monoxide, dealkylation of toluene with water vapor into benzene, and acidylation of diethyl amine with m-toluic acid (performed under optimum conditions with either conventional or microwave heating).

Parameters	Joint deep oxidation of n-C_4H_{10} and CO			Dealkylation of toluene with water vapor			Acidylation of diethyl amine with m-toluic acid		
	A	B	C	A	B	C	A	B	C
Temperature (°C)	250	230	230	430	430	420	370	370	370
Contact time (sec)	0.7	0.7	0.7	3.52	3.52	2.73	8.0	6.5	3.6
Conversion (%)	94.6	98.2	97.8	73.6	75.8	76.2	87.4	89.6	90.2
Selectivity (%)	~100	~100	~100	75.6	87.8	92.0	88.3	88.8	87.4
E_A (cal/mol)	20240	20235	20250	26200	26400	26300	121.8	119.6	120.4
lg k_0	6.470	9.414	9.133	6.470	8.614	9.213	6.323	8.770	8.485

'A' denotes catalyst synthesis and the reactions that occurred under conventional heating conditions. The reactions were conducted by conventional electric coil heating of the reactor; 'B' denotes the catalyst synthesized by microwave heating; 'C' denotes the catalyst synthesis and the reactions that were conducted by microwave heating.

Based on these results, we concluded that the fine-grained aluminum powder-reinforced γ-Al_2O_3/Al support of the active mass of heterogeneous catalysts synthesized by microwave heating showed significant advantages over similar support samples obtained by conventional heat treatment regarding textural properties and the microwave radiation absorption rate. As the support showed a high level of microwave energy absorption, some amount of energy was saved during its preparation and operation.

Due to these characteristics, this universal support can be recommended for producing supported catalysts to perform microwave field-stimulated reactions.

Author Contributions

The author did all the research work of this study.

Competing Interests

The author has declared that no competing interests exist.

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