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Ultrasound in Catalysis

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Abstract

The results of studied on the use of ultrasound in catalyst synthesis and processing are summarised. It is demonstrated that exposure to ultrasound can regulate the textural and structural characteristics of catalysts by varying exposure time and the amplitude of ultrasonic radiation. Optimisation of the conditions of ultrasonic treatment of the catalysts is determined to provide improvement of their physicochemical properties. This allows achieving higher reagents conversion and selectivity of the formation of target products in various chemical reactions. Examples of ultrasound application from the viewpoint of following the green chemistry principles are considered. It is shown that ultrasound application in organic synthesis promotes improvement of chemical process characteristics and reduces reaction time.

Keywords: ultrasonic treatment, ultrasonic cavitation, ultrasound in catalysis, ultrasonic catalyst synthesis, catalysis

Advance in industrial technologies is a cornerstone for successful resolution of the practical issues aimed at meeting human requirements. This is achieved by applying innovative and scientific approaches. The products of petrochemical synthesis are used in many areas, but new methods are necessary to enhance their output. One of the routes to solve the formulated tasks is in using physical methods for treatment and conversion of the substances and materials to improve the qualitative and quantitative parameters of the technological patterns of industrial processes.

There are many methods to treat catalysts to enhance their efficiency: irradiation within the ultraviolet [1] and infrared range [2], heating with the radio-frequency waves [3], the action of magnetic field [4] and ultrasound [5]. As early as at the end of the XIX century, the first studies of ultrasound were carried out by the Russian physicist P. N. Lebedev [5], which provided an impetus to the studies in this area.

Ultrasound is the elastic oscillations and waves with the frequencies above 20 kHz. Ultrasonic treatment involves the destruction of colloid particles at the micrometre level, which promotes the acceleration of chemical reactions due to acoustic cavitation. With ultrasound intensity more than 10^5 W/m^2 , the kinetic energy, formed due to collapsing bubbles in the liquid medium and concentrated in a negligibly small volume, is partially transformed into the force pulse and partially into thermal energy, which causes heating of the substrate under test. In liquid media, under the action of ultrasonic oscillations, electrokinetic phenomena also arise due to the directed motion of charged particles, and they affect diffusion processes. The arising sound wave can propagate in all the three phases. As a result, the deformation shift of particles or the distribution of some molecules in the interspatial system of other ones occurs in the substance [6].

The application of ultrasonic technologies opens the way to obtaining new substances and materials by changing their properties (for example, by using nanosized particles, by distributing solid substances in a liquid medium, diffusion of liquid phases, etc.) [5].

By means of ultrasonic treatment, the authors of [7] carried out the synthesis of nitrogen-doped TiO_2 catalyst supported on magnetically separable $\text{Fe}_3\text{O}_4/\text{ZnO}$ particles. Material preparation was performed with ultrasonic power variations from 80 to 120 W and treatment duration from 15 to 75 min. The optimal regime of treatment was determined: power 80 W, time 30 min. As a result, the minimal size of the obtained particles was 31.22 μm . For comparison, the size of catalyst particles obtained by traditional synthesis was 806.4 μm , which points to particle agglomeration in the absence of ultrasonic treatment. Both types of the catalyst were tested in the catalytic desulphurisation of thiophene. It has been established that the activity of catalyst synthesised using ultrasonic treatment is higher, which is explained by a decrease in particle size and attainment of system homogeneity. In general, the cited work demonstrates the advantages of ultrasound for the improvement of catalyst characteristics, as well as the successful application of catalysts obtained with the help of ultrasound for desulphurisation.

The authors of [8] presented comparative characteristics of TiO_2 catalysts doped with Fe(III) , synthesised using two different methods: ultrasound treatment and sol-gel. Investigation of the catalysts was carried out using a large set of physicochemical methods. Superiority of the catalyst synthesised through ultrasonic treatment was established, with the following parameters: specific surface area reached 49 m^2/g , and particle size was 99 nm. The results of forbidden gap measurement by UV-VIS spectroscopy demonstrated its decrease from 3.2 to 2.9 eV. To test catalyst activity, photocatalytic experiments were carried out, and they confirmed the superiority of iron-doped TiO_2 catalyst synthesised using ultrasound: this catalyst provides a higher degree of Acid Blue 80 dye decomposition (38 %) in comparison with the catalyst synthesised using the traditional method (31 %). So, ultrasonic treatment of the catalyst improves its activity in the process of Acid Blue 80 dye decomposition.

The authors of [9] synthesised the nanosized Pt-graphene oxide (OG)- TiO_2 photocatalyst using the ultrasonic method, and characterised the syn-

thesised catalyst. To evaluate the catalytic activity of Pt-OG- TiO_2 nanoparticles, photocatalytic and sonophotocatalytic decomposition of anionic surfactant dodecylbenzene sulphonate (DBS) in the aqueous solution was carried out. It has been determined that the Pt-OG- TiO_2 catalyst promotes DBS decomposition at a higher rate than P-25 (TiO_2) catalyst. The Pt-OG- TiO_2 catalyst demonstrated a substantial degree of DBS decomposition under the action of visible light. The acidity of the initial solution affected the rate of photocatalytic DBS oxidation, while this effect was not observed for sonochemical or sonophotocatalytic DBS oxidation.

New catalysts were obtained with the help of ultrasonic and plasma treatment: phosphotungsten heteropolyacids (PW_{12}) supported in aluminium oxide. The catalysts were characterised using various spectroscopic methods, and their catalytic properties were evaluated in the process of cationic polymerisation of tetrahydrofuran. Results of investigations show that plasma treatment causes a substantial increase in the acidity of catalyst surface, while ultrasonic treatment promotes the uniform distribution of PW_{12} over the surface of the support and release of a larger amount of active centres for the acid catalytic reaction. The activity of the synthesised catalyst (69.7 %) exceeds the activity of the catalyst obtained using the traditional method (57.5 %) [10].

Ultrasonic synthesis of new pyrazoles using Ni-Mg-Fe layered double hydroxides (LDH) as the catalyst of 1,3-dipolar cycloaddition was described. The proposed method was determined to lead to the high yield, short reaction time, and to obtain environmentally friendly solvent. Catalyst activity remains almost unchanged after 6-fold repeated use, so the indicated process can be considered as environmentally friendly. The smallest size of Ni-Mg-Fe LDH catalyst particles is equal to 29 nm [11].

The authors of [12] used mechanical action (milling in ball and planetary mills) and ultrasonic treatment of zeolite in water to synthesise nanoparticles from the industrial sample MFI type zeolite. It has been determined by means of diffuse reflectance IR spectroscopy, X-ray diffraction, NMR spectroscopy of solids, and chromatography that zeolite milling leads to partial destruction of its structure and the formation of defects in the crystal framework, while under an ultrasonic action in the aqueous medium the zeolite lattice is conserved completely. Under the action of ultrasound, MFI agglomerates are destroyed, and nanoparticles up to 40–50 nm in size are

formed. Ultrafine suspensions have been obtained by dispersing zeolite nanoparticles in highly boiling liquids – silicone or hydrocarbon oils (Syltherm 800 or Dowtherm RP, respectively). The stability of these dispersions depends on the type of dispersing medium. The nanosized suspensions of zeolite in Dowtherm RP are more stable than suspensions in Syltherm 800: the former are conserved without mixing for at least three weeks (with settling at room temperature).

Some mesoporous ZSM-5 zeolites were synthesised by ultrasonic treatment, and their activity in the catalytic cracking of light naphtha was investigated. The ZSM-5 zeolite was synthesised from rice husk ash without using any organic matrix, and its properties were studied by physicochemical methods. It has been discovered that ultrasonic energy promotes the formation of the hierarchic structure of ZSM-5 during alkaline treatment. According to the results of X-ray diffraction analysis, zeolite structure was conserved after ultrasonic treatment for 20 min in an alkaline medium. However, long-term desilication causes destruction of MFI zeolite structure, and roughness appears on zeolite surface with an increase in the duration of alkaline treatment. The synthesised ZSM-5 has a hexagonal structure with highly ordered morphology. Results of the tests of catalytic activity show that ultrasonic energy has a substantial effect on the activity of ZSM-5 [13].

As described in [14], a nanosized metal catalyst containing hemin supported on technical carbon (carbon black) for oxygen reduction was synthesised. The catalyst was subjected to thermal and ultrasonic treatment to increase the surface area (BET) and to provide a larger number of available active centres. The catalyst was characterised by physicochemical methods of analysis. It is shown that its catalytic activity in oxygen reduction increases substantially due to a decrease in particle size and an increase in the surface area.

The authors of [15] prepared two types of catalysts: annealed Fe/Co/Al-Mt(C) and ultrasound-treated Fe/Co/Al-Mt(U) – using a traditional ion exchange method and in combination with ultrasound, where iron was the active component, while montmorillonite (Mt) was used as a support. The physicochemical properties and surface structure of (Fe/Co/Al-Mt) samples were studied by IR spectroscopy, X-ray diffraction, UV-Vis spectroscopy, scanning electron microscopy. The efficiency of thus obtained catalysts was tested in the process of waste water purification. It has been de-

termined that in comparison with Fe/Co/Al-Mt(C) the specific surface of Fe/Co/Al-Mt(U) increased by 16.2 %, pore diameter increased by 10.2 %, and the active component was dispersed more uniformly. The Fe/Co/Al-Mt(U) catalyst obtained with the help of ultrasound demonstrated higher stability and lower loss of the active component: after four tests, the activity of this catalyst was still higher than 70 %, and elution of iron ions from the active component was only 0.59 mg/L.

The authors of [16] synthesised the catalysts from solid industrial wastes (slag from desulphurisation setup) for carboxymethylation. Initial slag material containing CaCO_3 , Ca(OH)_2 , SiO_2 , Al_2O_3 , Fe_2O_3 and TiO_2 was treated with various reagents, and synthesis parameters were varied. To enhance slug dissolution and crystallisation of the new phases, ultrasonic treatment was used. The physicochemical properties of initial materials and synthesised catalysts were evaluated using several analytical methods. Ultrasonic treatment of industrial slag possessing low specific surface ($6 \text{ m}^2/\text{g}$) and weak crystal structure caused the formation of highly crystallised catalyst with surface area up to $49 \text{ m}^2/\text{g}$. The catalytic activity of the obtained catalyst was studied in the synthesis of esters by carboxymethylation of cinnamic alcohol with dimethylcarbonate at 150°C , and the catalyst demonstrated high selectivity with respect to the target product (about 84 %). So, ultrasonic treatment had a positive effect on the catalytic activity of the synthesised catalyst.

The authors of [17] synthesised the nanoparticles of copper (I) sulphide (Cu_2S) by thermal decomposition of diethyldithiocarbamate complex of copper (II) at 220°C . Analysis of the synthesised product was carried out using various spectroscopic and physical methods, and confirmed the formation of pure Cu_2S composed of nanoparticles about 50 nm in size. To decompose methylene blue dye over Cu_2S nanoparticles, an improved oxidation process was developed (with H_2O_2 as an oxidiser) using ultrasound. It has been determined that the parameters of the process involving ultrasound are improved in comparison with catalysis without ultrasound. It is shown that synergism between Cu_2S catalyst and ultrasonic waves depends on catalyst dosing and H_2O_2 concentration.

In [18], evaluation of the effect of ultrasound on the disperse characteristics of magnesium hydroxide was carried out. It has been determined that particle size is affected by the concentrations of initial reagents, duration and intensity of ultrasonic action. The source of ultrasound was

IL 100-6 tool operating at the working frequency of 22 kHz and the adjusted oscillation amplitude within 0–80 μm . The studies of particle size revealed that the efficiency of ultrasound decreases with an increase in the volume of the medium under treatment. This may be due to the power of the tool, and with shock waves that are to destroy the aggregates of particles but do not destroy them actually because of the large volume of the medium under treatment. So, no less important role of ultrasonic power was demonstrated by this result.

The authors of [19] studied the kinetics of phenol sulfoxidation in the presence of cobalt, nickel and iron complexes supported on the polymeric matrix (polyacrylic acid, PAA) in the ultrasonic field within a frequency range of 0–200 kHz at a power of 5 W/cm³. The conditions of ultrasonic action were optimised. It is shown that ultrasound within the studied range causes enhancement of the oxidation-reduction potential of the system MX–PAA–C₆H₅OH–H₂O (MX = salts of Co²⁺, Ni²⁺, Fe²⁺) to 200–400 mW, promotes the formation of binuclear complexes of cobalt and nickel at lower salt concentrations in solution, leads to an increase in the constants of complex formation for cobalt, nickel and iron. It is determined that ultrasound affects the nature of intermediate complexes and, as a consequence, on their activity in sulfoxidation process.

The authors of [20] demonstrated the ability of ultrasound to improve the catalytic properties of a solid catalyst. In addition to enhancement of heat and mass transfer caused by collapses of cavitation bubbles, the authors demonstrated synergistic effect between the catalysis and ultrasonic treatment. In particular, physical and chemical local action on the catalytic surface during the collapses of cavitation bubbles can lead to metal ion oxidation, provide energy to the catalyst/reaction, activate or form catalytic centres *in situ* and even present catalyst deactivation. The effect of ultrasound on the structures of solid catalysts that may have positive or negative influence on catalytic characteristics is discussed.

The authors of [21] studied the effect of ultrasonic treatment on the catalytic properties of manganese-containing catalyst based on nanostructured carbon material obtained by liquid-phase oxidation of naphthene-paraffin concentrate. Ultrasonic treatment was determined to promote improvement of the catalytic properties and to accelerate hydrocarbon oxidation to syn-

thetic petroleum acids, in addition, it allows the repeated use of the catalyst in reactions.

Oxidation of *n*-undecane in the presence of the synthesised catalyst (chromium salt of natural petroleum acid (RCOO)₃Cr) treated with ultrasound is described in [22]. The distribution of catalyst particles over the liquid phase before and after ultrasonic treatment in hydrocarbon medium was determined by the dynamic light scattering. As a result of ultrasonic action, the average size of catalyst particles shifts from ~7 to ~15 nm. A mixture of the catalyst with hydrocarbon was treated with ultrasound and then subjected to liquid-phase aerobic oxidation in the oxidative installation of bubbling type. Ultrasound was determined to promote acceleration of the formation of oxygenated products, reducing reaction time by a factor of 2. The yield of final products increases by a factor of 12 in comparison with autocatalysis and by a factor of 2.5 in comparison with catalysis without ultrasound.

The authors of [23] applied treatment with ultrasound in the process aimed at enhancement of the strength of industrial cracking catalyst Lyuks. The effect of ultrasonic treatment on dispersity, strength, hydrophilicity and phase composition of the components of cracking catalyst and the catalyst composition in general was studied. The composition of commercial cracking catalyst includes montmorillonite, the product of thermochemical activation of silica, amorphous aluminosilicate and zeolite Y. The application of ultrasonic treatment to the suspensions of separate components of the catalyst composition causes an increase in their dispersity, an increase in hydrophilicity of montmorillonite particles, change of the rates of phase transitions in aluminium hydroxide, an increase in the strength of the extrudates of annealed montmorillonite from 200 to 300 kg/cm². Ultrasonic treatment of a suspension of cracking catalyst suspension leads also to an increase in its dispersity, thus increasing its strength from 80 to 125 kg/cm² and the bulk density of final cracking catalyst, with the conservation of high catalytic activity without any changes in its composition and without introducing additional stage – peptisation, which is traditionally used for these purposes.

A multifunctional basic catalyst based on tungsten-decorated carbon quantum dots (CQDs), A-CQDs/W, has been developed in [24] as an attempt to implement the idea of environmentally friendly catalytic oxidation of alcohols into cor-

responding aldehydes, and the efficiency of this catalyst has been investigated in sono-oxidation in the presence of hydrogen peroxide, used as environmentally safe oxidiser in aqueous medium. The key role of ultrasonic treatment in achieving the high yield of the target product in the test reaction of benzyl alcohol oxidation was demonstrated. As a result of the action of acoustic waves, exclusive temperature conditions are created, along with pressure conditions providing the high extent of reagents interaction. As a result, the maximal product yield is achieved within a relatively short time, which, in turn, leads to a decrease in the amount of unreacted reagents and by-products. This completely eliminates the necessity to use toxic organic solvents and dangerous oxidisers, auxiliary substances and the catalysts of inter-phase transport.

Green chemistry becomes increasingly widespread due to improvement of technological processes having a positive effect on the environment. Ultrasound is one of the important methods to improve organic synthesis from the viewpoint of green chemistry, because it can promote an increase in the yield and selectivity, and reduction of reaction time in comparison with traditional methods [25].

Biocatalysis (enzymatic catalysis) is the area of active development at present. The use of enzymes (biologically active substances) as the catalysts accelerating the synthesis of carbon-based compounds becomes a useful alternative to traditional chemical catalysts because biocatalysis relates to the area of green production of chemical products. However, the processes of enzymatic nature usually provide lower yields in comparison with usual chemical processes. For this reason, a broad application of ultrasound in enzymatic processes is observed during the recent years, in particular in the production of esters with the desired characteristics for pharmaceutical, cosmetic, and food industry, hydrolysis and glycerolysis of vegetable oils, biodiesel production, etc. It is indicated in some studies that the energy released by ultrasound during cavitation can be used to enhance mass transfer (substrate/enzyme), thus increasing the rate of product formation and promoting an increase in the catalytic activity of enzymes. In addition, ultrasonic treatment is considered to be a green technology because of its high efficiency, low requirements to instrumentation, and substantial decrease in treatment time without the introduction of additional chemical reagents. The authors of [26] generalised the studies

aimed at ultrasound application in enzyme-catalysed reactions: esterification, hydrolysis, glycerolysis and re-esterification [26].

A comprehensive review of achievements in the methods of organic synthesis reported within the past decade is presented in [25], with a focus on multicomponent reactions involving ultrasound under the conditions of heterogeneous catalysis. In particular, pharmacologically significant N- and O-heterocyclic compounds are considered, the methods of their synthesis using green solvents and catalyst recycling are described. Heterogeneous catalysis is one of the essential aspects of sustainable chemistry, with its major advantage – recycling and repeated use of the catalysts. On the other hand, multicomponent reactions provide the synthesis of structurally diverse compounds in a single reactor without isolating and purifying the intermediate products. So, a combination of ultrasound with heterogeneous catalysis turned out to be a powerful tool to obtain compounds spending less time and energy.

CONCLUSION

Numerous studies carried out during the recent years show that ultrasonic treatment is an efficient method to prepare, modify and/or process catalysts for the purpose of improving their catalytic properties. Ultrasonic action can be used to regulate physicochemical and functional characteristics of catalysts by varying the time of action, power and amplitude of ultrasound. Optimisation of the conditions of ultrasonic treatment provides improvement of catalyst properties. In particular, it allows achieving high reagent conversion and selectivity of the formation of target products.

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