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THEORETICAL FUNDAMENTALS OF MECHANOCHEMICAL REACTIONS IN THE SOLID PHASE

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Abstract: Chemical reactions in the solid phase are characterized by many specificities. The study of such reactions, with regard to the condensed state, eliminates the need to introduce many approximations that are common when considering reactions in the liquid phase or heterogeneous reactions, other laws, specific and inherent only to the solid state of matter. These make the scientific field of solid-state chemistry far more complex, due to the very strong need for a comprehensive multidisciplinary approach. The basic theoretical principles from the fields of physics, chemistry, physical chemistry and chemical technology are equally important. The article gives an example of mechanochemical synthesis of the neutralization type, and barium oxide as the base component and titanium oxide as the sisal component of the mixture are activated. In order to determine the changes that occur due to the mechanical activation of the starting materials, barium oxide and titanium oxide were ground in a stoichiometric ratio for 440 minutes. The analysis determined that a mechanochemical synthesis had taken place and that barium titanate with a degree of synthesis of 0.99 had been obtained. This was confirmed using X-ray structural analysis, which monitored the state of the activated material as a function of the time of activation.

Keywords: solid state, mechanochemical synthesis, barium titanate

1 INTRODUCTION

The possibility of achieving a chemical reaction between the reacting components of solid substances, without the phase of their dissolution and decomposition into ions, has always been attractive to chemists and encouraged constant research in the field of solid-state chemistry and physics. Regardless of whether the reaction is thermodynamically possible or not, the problem that needs to be overcome in initiating such reactions is

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initiating the beginning of the reaction, i.e. bringing the required amount of energy that will be equal to or greater than the required activation energy for that reaction and the conditions in which it should occur. takes place. For this reason, methods of realizing such reactions using thermal activation were mainly researched and developed. The method of mechanical activation, which also enables the creation of active (excited) and unstable states in a solid material, reveals completely new perspectives for accelerating such reactions. Therefore, it is possible to define several methods of application of mechanical activation for this purpose (Feng, Fahrenholtz and Brenner, 2021; Sani et al., 2017; Fang et al., 2018; Đorđević et al., 2022; Obradović et al., 2015):

- previous individual mechanical activation of the input components (or one of them) before thermal activation,
- previous simultaneous mechanical activation of the mixture of input components before thermal activation,
- realization of a chemical reaction directly during the communication of mechanical energy to the reaction system (mechanochemical method),
- realization of a chemical reaction by simultaneous application of mechanical and thermal activation (mechanothermic method).

Mechanochemical methods of conducting reactions in mixtures of solid materials, as well as methods of previous simultaneous mechanical activation, are extremely important for this issue.

In accordance with the basic principles of chemical thermodynamics, the direction of the chemical reaction and the degree of mutual reactivity of the starting reactants are determined by the isobaric-isothermal potential of the reaction ΔG (Wang et al., 2016; Pandey, Buljak and Balać, 2017). A reaction between chemical elements and compounds in the solid state is possible if:

$$\Sigma\Delta G_{\text{products}} - \Sigma\Delta G_{\text{reactants}} = \Delta G_{\text{reactions}} < 0 \qquad (1)$$

For reactions of the type:

$$A_{ss} + B_{ss} \rightarrow C_{ss} + D_{ss} \text{ (or } AB_{ss}) \qquad (2)$$

(ss-solid state)

$$\Delta G_{\text{reactions}} = \Delta G^{o}_{\text{reactions}} + RT\ln(a_C a_D / a_A a_B)$$

where aD, aC, aA, aB - activities of reaction products and reactants.

If solid substances are exposed to normal pressure and if their activities are equal or close to unity, then it is:

$$\Delta G_{\mathrm{reactions}} = \Delta G^{o}_{\mathrm{reactions}} \tag{3}$$

To determine the direction of the reaction, it is sufficient to know whether the value of ΔG of the reaction at standard conditions ($T=298°K$, $p=0.1$ MPa) is positive or negative. If the reaction is thermodynamically possible ($\Delta G < 0$), then in a mixture of solid reactants it will theoretically go to the end.

If a liquid or gaseous phase appears as a product of the reaction or solid solutions are formed, then the influence of the term $T\Delta S$ in the expression for the change in the isobaric-isothermal potential

$$\Delta G = \Delta H - T\Delta S \tag{4}$$

becomes very expressed, and then it is difficult to talk more about the ratio between the direction of the reaction and the sign of the ΔG value, because the entropy change in such processes can be both positive and negative. During the mechanical activation of solid materials, transformations and accumulation of released mechanical energy occur in the form of crystal lattice defects, displacement of atoms from their equilibrium positions, etc., which leads to an increase in enthalpy and entropy of the treated system. In a large number of cases, this change is significant, and then the change in free energy can be such that the reaction changes its direction. Insufficient study of those factors can lead to the conclusion of the alleged inconsistency of the obtained results with thermodynamic calculations (Ewais et al., 2017).

The characteristic features of chemical reactions in the solid phase are their progress in stages and the localization of the chemical process, which takes place only at the points of direct contact of the starting reactants. The following stages have the greatest influence on the kinetics of chemical processes in the solid phase (Kerbart et al., 2019): communicating the required amount of energy to initiate and start a chemical reaction, creation of the first nuclei of the chemical reaction product and diffusion of reactants through the layer of the resulting product.

The chemical reaction begins first on the most active parts of the surface of solid substances, namely the beginnings of dislocations, point defects of the crystal lattice, etc. This is where nuclei of chemical reaction products are formed, whose growth and fusion lead to the creation of a compact reaction zone. Diffusion transfer of reactants through the resulting product layer represents a slow step in the further course of the reaction and can take place according to different mechanisms. In any case, by acting on the slow stage of the process, it is possible to act on the speed of the chemical reaction in the solid phase. Mechanical activation of the mixture of solid reactants is one of the most effective ways to eliminate the interference during the reaction caused by the diffusion of reactants through the layer of the resulting product.

In order to understand and clarify the phenomena that accompany mechanochemical reactions, it is necessary to monitor and clarify the factors that make mechanochemical reactions specific. These factors are primarily the local pressure and temperature that occur at the points of collision of reactants with grinding bodies, as well as specific phenomena that occur during plastic flow of materials. Thus, the impulsive nature of local temperatures can give a mechanochemical reaction a completely different course compared to reactions between the same reactants that take place with gentle heating. When multiple parallel or subsequent reactions are possible in the combination of two or more reactants, only those that are thermodynamically possible in a short-term thermal impulse at the moment of collision will occur during mechanical activation (Marc et al., 2013). In addition, when it is taken into account that most of the mechanical energy during mechanical activation is transformed into thermal energy, it is clear that heat has a significant impact on the course and outcome of mechanochemical reactions.

1.1 Mechanochemical syntheses

Mechanochemical synthesis refers to chemical reactions that take place in the solid phase, initiated by mechanical energy input, in mechanochemical reactors (special mills, disintegrators, etc.), and in which one or more reactants are obtained from two or more reactants, according to a clearly defined chemical reaction new substances - reaction products. Figure 1 schematically shows the transformations during the mechanochemical process (Raghu, Nampoothiri and Satish Kumar, 2018).

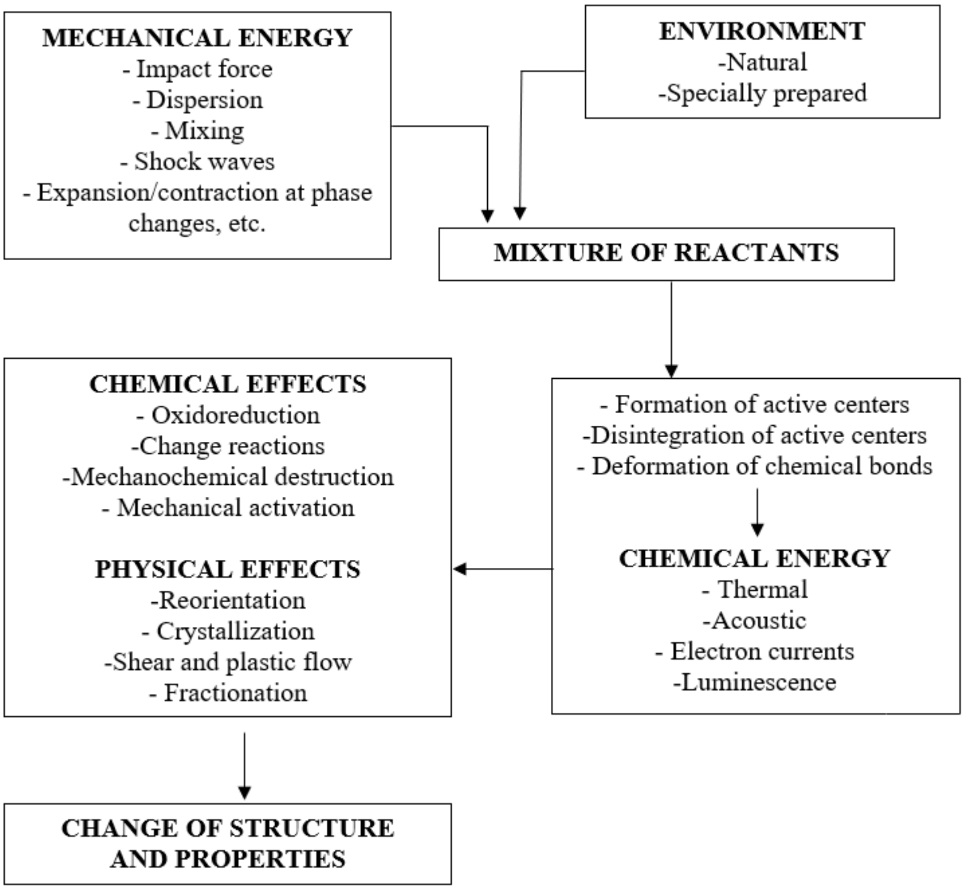

Figure 1 Schematic view of mechanochemical transformations

It is noticeable that during the mechanochemical process, in addition to the chemical reaction, a number of other transformations take place, which directly or indirectly affect the course and result of the chemical reaction. If the subject of scientific study is precisely the chemical reaction that takes place in the solid phase during the mechanochemical process, then the study of other, parallel, effects and transformations is aimed at elucidating and defining the kinetics, mechanism, and other essential features of the mechanochemical reaction.

Considering all forms of chemical reactions in general, it is most correct to divide them into two general groups: chemical reactions in which oxidation-reduction changes occur in elements that are part of the reacting chemical compounds and reactions in which such changes do not occur. However, for the study of mechanochemical reactions, it is necessary to make a more detailed division that more clearly specifies the final outcome of a mechanochemical reaction. In this way, it is possible to more clearly specify the conditions under which the mechanochemical process will take place, and thus it is easier to control and manage parallel effects that decisively affect the parameters of the microenvironment in which the chemical process takes place. In this regard, perhaps the most appropriate division of mechanochemical reactions into (Ewais et al., 2017): neutralization reactions, ion exchange reactions, union reactions i redox reactions. It has been shown in previous research (Marc et al., 2013) that this division enables a fairly precise division of the parameters of mechanochemical processes, as well as the division of parameters related to the choice of reactants (thermodynamic parameters and other physical and chemical characteristics of reacting substances and possible reaction products).

1.1.1 Mechanochemical reactions of neutralization

If one strictly starts from the basic principle of mechanochemistry, that mechanochemical reactions take place exclusively in the solid phase, then mechanochemical reactions of neutralization could be called those chemical reactions that take place under the influence of mechanical energy between oxides-anhydrides of acids and oxides-anhydrides of the corresponding hydroxides, with the condition that both substances are in a solid state under normal conditions (Pandey, Buljak and Balać, 2017). Such, for example, are the reactions between WO_3, MoO_3, SiO_2, TiO_2, V_2O_5, Fe_2O_3, etc. as one reactant, (anhydrides of corresponding acids) and BaO, CaO, MgO, SrO, NiO, CoO, etc. as a second reactant (oxide bases of corresponding hydroxides). (Lazarević et al., 2009; Li and Shih, 1997).

Such reactions take place according to the general equation:

$$Me^{I}O + Me^{II}O_n = Me^{I}Me^{II}O_{n+1} \tag{5}$$

where n = 2, 3, etc.

However, it is advisable to expand the list of potential reactants for mechanochemical neutralization reactions with the following groups of reactants: all hydroxides, acids that under normal conditions are in a solid state and carbonates, formed from basic oxides and corresponding hydroxides (Raghu, Nampoothiri and Satish Kumar, 2018).

Regardless of the fact that hydroxides and acids contain constitutional water, which in their mutual reaction will certainly be a product of the reaction, thus potentially introducing a liquid phase into the reaction system, it is also a fact that a large number of salts, formed in neutralization reactions, crystallize together with water, that is, in their crystal structure, and thus in their chemical composition, they contain a certain number of water molecules. Crystalline water constitutes that substance by its physical and chemical characteristics, which is essentially different from the same but anhydrous substance. When it comes to carbonates, there are two important reasons that make them suitable for being classified as reactants in mechanochemical neutralization reactions:

- the carbonate ion, as the base of extremely weak carbonic acid, allows carbonates to retain most of the properties of hydroxides and basic oxides from which they were formed;
- the product of the mechanochemical reaction of neutralization, in which the appropriate carbonate participates instead of hydroxide or basic oxide, will be gaseous carbon dioxide, which, if the process takes place in an open system, will immediately leave the reaction zone, so that it will not interfere with the further flow and outcome of the reaction.

Bearing all this in mind, as well as the results of previous research, it can be assumed that mechanochemically activated substances will react with each other in the solid phase by the mechanisms of neutralization reactions and in the case of binary systems of the general type: acid/acid anhydride + metal (hydr)oxide/metal carbonate (Lazarević et al., 2009; Li and Shih, 1997; Avvakumov, Senna and Kosova, 2001; Đorđević, Vlahović and Mihajlović, 2023).

Neutralization reactions in mechanochemical syntheses are interesting for research, and one of these reactions is the preparation of bismuth titanate from bismuth and titanium oxides. In the article of the group of authors (Lazarević et al., 2009), mechanochemical activation of a stoichiometric powder mixture $2Bi_2O_3$ and $3TiO_2$ was performed, and the compound $Bi_4Ti_3O_{12}$ was obtained. Powder X-ray diffraction was used to monitor the intermediate product as well as the product obtained after the optimal activation time. The authors investigated the mechanochemical activation of the mixture $2Bi_2O_3$ and TiO_2, where the activation was performed in a planetary ball mill (Li and Shih, 1997). To monitor the parameters (change in crystallite size, the presence of an amorphous phase and the product obtained by transforming the initial components, they were monitored by the method of X-ray diffraction analysis. The authors of the paper (Li and Shih, 1997) established that there was deformation of the crystal lattice and destruction of the structure of the starting material. After a certain amount of grinding, the analysis showed that they obtain highly disordered, amorphous/nanocrystalline structure.

1.1.2 Mechanochemical reactions of ion exchange

Studies of mechanochemical reactions of ion exchange have shown that this type of reaction, when it takes place in the solid phase, strictly obeys the laws of chemical kinetics and chemical thermodynamics Namely, it was established that the exchange of ions in a mixture of two chemical compounds (salts) occurs kinetically exactly as it can be predicted, considering the strength of the acids or bases, from which the reacting salts were formed (Avvakumov, Senna and Kosova, 2001).

1.1.3 Mechanochemical oxido-reduction reactions

This type of mechanochemical reactions, considering the nature of possible reactants, is reduced mainly to reactions of metal oxides with elemental metals, non-metals and metal sulfides (Gonzales et al., 2017). All these reactions can be represented by the following general equations:

$$Me^{I}O_{n/2} + n/mMe^{II} = Me^{I} + Me^{II}{}_{n/m}O_{n/2} \qquad (6)$$

$$MeO_{n/2} + n/4xC = Me + n/4xCO_2 \qquad (7)$$

$$MeO_{n/2} + n/4xSi = Me + n/4xSiO_2 \qquad (8)$$

$$MeS + 2\,MeO = 3Me + SO_2 \qquad (9)$$

$$Me^{I}S_2 + Me^{II}O_2 = Me^{I}S + Me^{II}O + SO_2 \qquad (10)$$

In Eq. 6, n and m are the degrees of oxidation of metals Me^{I} and Me^{II} in the corresponding oxides, respectively. In Eq. 7 and 8, n is the degree of oxidation of metal Me in the corresponding oxides. It has been shown that these reactions, when they take place in the solid phase, obey to a large extent the predictions that can be made based on the thermodynamic parameters of the corresponding reactions.

1.1.4 Mechanochemical reactions of compounding

Mechanochemical joining reactions have been the most frequently studied so far, and there are the most achieved results in that area. This statement refers both to the number of processed and tested combinations of reactants, as well as to the study of the kinetics and mechanism of the process and the characteristics of the obtained products. The most numerous groups consist of works in which the possibility of mechanochemical production of substances such as complex oxides, carbides, nitrides and hydrides is studied. Different possibilities of obtaining were examined, and two directions were mostly considered: previous individual mechanical activation of the starting substances and simultaneous mechanical activation of those substances that participate in the construction of the final material. The essence and goal of mechanochemical joining reactions is to obtain substances with specific physical and especially crystallographic characteristics. Namely, all these works are carried out with the aim of obtaining materials for specific purposes: ceramics, metal-ceramics, glass, metal-glass, superconductors, additives for alloying, etc. In the mechanochemical process of joining, the entire transformation during the process takes place between the reacting particles of the nanometer-sized particles, and the specific properties of the obtained material result primarily from the phenomena that occur on particles of these sizes. For this reason, in recent years, the terminology in this field has been adapted to this knowledge, so often the materials obtained in mechanochemical reactions of union are called nanomaterials, nanopowders, nanocrystalline powders, etc. (Kavairasu and Ravichandran, 2020; Zhang, et al., 2020).

2 EXPERIMENTAL PART

The authors of this paper are involved in experimental research in the field of mechanochemical activation and mechanochemical synthesis. Therefore, here we will present the results obtained from the solid-state synthesis process, specifically neutralization reactions in the mechanochemical synthesis reaction. The aim was to support the theoretical considerations of the basis of neutralization reactions presented in the introductory, theoretical, part of the paper with concrete results.

2.1 Materials and methods

For the experimental research presented in the paper, the starting components barium oxide BaO, as a basic reactant and titanium dioxide TiO_2 as an acid, were used. Barium oxide manufactured by FLUKA (Switzerland), CAS No. was used in the experiments. [1304-28-5], quality pure and TiO_2 produced by MERCK (Germany), CAS No. [13463-67-7], pro analysis quality.

Recalculated according to reaction 6, the amount of starting substances consisted of 0.5 mol (76.7 g) BaO and 0.5 mol (39.9 g) TiO_2, so the total amount of starting material amounted to 116.6 g.

$$BaO(s) + TiO_2(s) = BaTiO_3(s) \text{ (or } BaO{*}TiO_2) \tag{11}$$

Chemical analysis. Chemical analysis was conducted using a Perkin Elmer Analyst 703 Atomic Absorption Spectrophotometer (AAS).

Procedures of mechanochemical synthesis. The mechanochemical synthesis of BaO and TiO_2 was performed in a high-energy vibration mill with torsion springs and annular working elements, manufacturer KHD Humboldt Wedag A.G., Germany, type MH954/3. The starting powders were activated for 440 minutes. The motor power of the mill used was 0.8 kW.

During the mechanochemical synthesis, samples were taken at 30, 110, 200, 290, and 440 min of activation, and X-ray structural analysis were performed. In this way, both starting components and products of mechanochemical processes are identified and monitored.

X-ray analysis. The samples were recorded and analyzed diffractometrically on an automated diffractometer with a copper tube PHILIPS PW-1700, which operates at 40 kV and 35 mA. The device is supplied with a graphite monochromator and a proportional counter, filled with xenon. The imaging angle (2θ) from 4 to 15° was used.

3 RESULTS AND DISCUSSION

3.1 Characterization of starting components

The starting components, BiO and TiO_2, which were used in the neutralization reaction were chemically analyzed and X-ray analysis was performed on the samples. X-ray diffractograms have been shown in previously published works, so only characteristic peaks are tabulated in this paper. They are important for monitoring the synthesis and formation of a new compound through the neutralization reaction that the authors wanted to show in this paper (Đorđević et al., 2023).

Table 1 shows the chemical characteristics of barium oxide.

Table 1 Chemical characterization of barium oxide

Content (acidimetric), min %	98
Substances insoluble in HCl, max %	0.005
Impurities, max%	
Carbonates (as CO_2)	0.5000
Chlorides (Cl)	0.0050
Sulfates (SO4)	0.0010
Total Nitrogen (N)	0.0020
Arsene (As)	0.0001
Calcium (Ca)	0.0200
Iron (Fe)	0.0050
Potassium (K)	0.0050
Sodium (Na)	0.2500
Lead (Pb)	0.0010
Loss on ignition, max %	0.5

The previously performed X-ray analysis confirmed the crystal structure of BaO and identified two characteristic peaks at certain wavelengths, which is shown in Table 2.

Table 2 Characteristic diffraction maxima of BaO

Pick no.	d-value	Angle 2Θ
1	3.2895	27.085
2	2.6771	33.445

Table 3 shows the chemical characteristics of titanium dioxide, while Table 4 shows the characteristic peaks obtained by X-ray diffraction, which confirmed its crystal structure.

Table 3 Chemical characterization of titanium dioxide

The content, %	99.0-100.5
Substances soluble in water, max %	0.5
Substances soluble in HCl, max %	0.5
Impurities, max%	
Heavy metals (like Pb)	0.0020
Acid-soluble antimony (Sb)	0.0002
Sulfates (SO_4)	0.0010
Total nitrogen (N)	0.0020
Acid-soluble barium (Ba)	0.0002
Arsenic (As)	0.0001
Iron (Fe)	0.0050
Acid-soluble lead (Pb)	0.0010
Zinc (Zn)	0.0005
Loss on ignition at 800°C, max%	0.5
Loss on drying at 105°C, max%	0.5

Table 4 Characteristic diffraction maxima of TiO_2

Pick no.	**d-value**	**Angle 2 Θ**
1	3.5133	25.330
2	2.3778	37.805
3	1.8926	48.035
4	1.6667	55.055
5	1.4806	62.700

3.2 Results of mechanochemical synthesis

The neutralization reactions between Bao and TiO2 and the formation of barium titanate were monitored by X-ray analysis of samples taken at precisely determined time intervals. (30, 110, 200, 290, and 440 min).

Figure 2 shows a comparative overview of X-ray structural analyses of the mechanochemical synthesis of barium titanate during activation.

The broadening of diffraction lines and their insignificant intensity indicate that after 30 minutes there was a significant disruption of the crystal structure of the initial reactants, especially titanium dioxide, whose crystal structure was very clearly expressed before the start of the reaction (Figure 2). The analysis of the registered peaks and corresponding diffraction angles confirmed the presence of initial reactants, but also the beginning of the formation of the product-barium titanate under some of the characteristic diffraction angles for this substance.

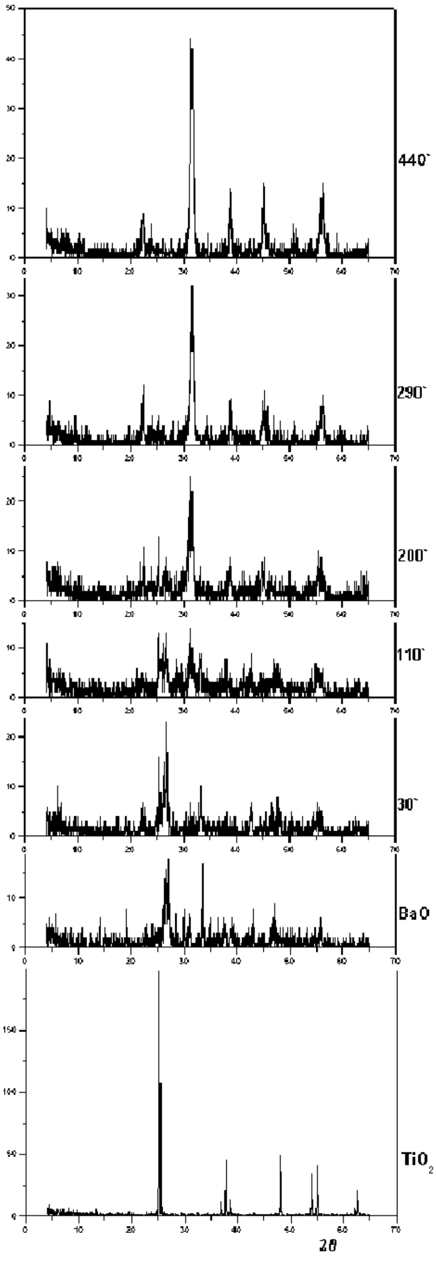

Figure 2 Comparative review of X-ray structural analyzes of the mechanochemical synthesis of barium titanate

The first phase takes place in the first 30 minutes of activation of the starting components and is the phase of progressive destruction of the crystal structure of the reactants. This part of the research has already been presented in the paper (Đorđević, Vlahović and Mihajlović, 2023). After the 30th minute of activation, subsequent X-ray structural analyzes showed that the characteristic peaks for the initial components are increasingly lost, and new ones are created (assuming that these are intermediate compounds).

It can be noted that the mechanical energy introduced into the reaction system, in this level, is mainly spent on the destruction of the crystalline structure of the starting reactants and their significant amorphization. The mechanochemical activation of individual substances (as well as the combination of two or more substances) leads to the transformation of the released mechanical energy and its accumulation in the treated material in the form of accumulated deformations of the crystal lattice, extremely developed and active specific surface of the material, whereby the material is brought to an elevated level of energy content. Further activation provides by introducing energy into the reaction system, which overcomes the energy barrier on the reaction path to the reaction product, so the chemical reaction irreversibly flows towards the expected product.

The final result of the X-ray structural analysis showed that after 440 minutes of activation, we obtained the final product, barium titanate, with a degree of synthesis of 99% (η=0.99). Initial components were not detected in the traces either, and further activation in the vibro mill did not lead to new changes in the structure. This results in an increased degree of potential energy, i.e. chemical reactivity of the material. Considering that the reaction involved pure and unactivated reactants that exist as elementary particles (BaO and TiO_2 molecules) as reactants during the mechanochemical reaction, it is concluded that the reaction of the mechanochemical synthesis of barium titanate proceeds in two phases. The first phase is mutual collisions between the molecules of two reactants, during which an activated complex is formed, which stoichiometrically correspond to the chemical composition of barium titanate, but in terms of structure and characteristics of chemical bonds, it represents a special compound.

4 CONCLUSION

Chemical reactions in the solid phase are specific in relation to other chemical reactions and different chemical laws apply to them, which are characteristic only for this aggregate state. Considering that the reactants remain in the solid phase, the action of the particles that enter the reaction is much more complex and requires a multidisciplinary approach. A particularly interesting area of solid-state reactions is mechanochemical synthesis, which depends on many input parameters. In addition to the input components, the device in which the synthesis process takes place, the type of activator, and the energy invested in the activation and synthesis process are also of great importance. Modern methods of monitoring the processes that take place in such devices, the application of modern scientific achievements in the fields of mechanics, crystallography, chemical thermodynamics, chemical kinetics, physical electronics, help to understand the complex processes that occur in activators.

As an example of mechanochemical synthesis in neutralization reactions in the solid state, an example of obtaining barium titanate from the initial components barium oxide and titanium oxide, where barium oxide (BaO) was used as a basic reactant, and titanium dioxide (TiO_2) as an acidic reactant, is given. The experiments showed that after 440 minutes of activation, the initial components were completely synthesized into the final barium titanate product, with a utilization rate of 99%. X-ray structural analysis after this activation time did not detect traces of the initial components, but only the final product.

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