Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Principles of Solid State Synthesis Method and Their Applications For Inorganic Synthesis

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Abstract

This paper discusses the principle of solid state synthesis method including definition, steps of reaction, mechanism of crystal growth, advantage and disadvantage, influencing factors, and tools. This paper also presents the examples of inorganic material synthesis such as metal oxides, metal salts, silicon carbide, zeolite, carbon, kaoline, and their composites by solid state method. The metal oxides were prepared at high, low, and room temperature, with conventional furnace and microwave, for single crystal, polycrystalline, and glass (amorphous material). Usage of microwave in synthesis of $MgAl_2O_4$ increased the crystallinity (1.5 X larger) and S_{BET} (4.4 X larger) and reduced the solid particle size (2.56 nm smaller) also synthesis time (5 h shorter) than usage of conventional furnace. The carbon was prepared by dry pyrolysis of various plant biomass, flours, coffee, and tea. The modified kaoline was made through substitution and intercalation. The carbon composites were created by impregnation of the CNS (Carbon Nanostructure) or CNM (Carbon Nanomaterial) using spinels. CNM or CNS solid particles size was conditionized by dispersion of the activated carbon in the water using mixer, blender, stirrer, and sonicator.

Keywords: Inorganic materials: inorganic synthesis; solid state method

1. Introduction

Synthesis is important because it is related to production of the new materials by using chemicals from laboratory experiments until industrial production [1]. Inorganic material is classified as single crystal, ceramic (polycrystalline) and glass (amorph). The single crystal is a solid which all its body is a one crystal, whereas the ceramic consists of multi crystals with particle size of from micron to ten microns, and glass is a solid with random atoms arrangement[2]. Crystal growth depends upon the material characteristics, such as melting point, volatility, solubility, and so on. Therefore there are four ways of crystal growth including from solution, from melt, from solid, and from vapour [3]. Research synthesis is a set of methods which integrate the individual empirical studies [4]. Classification of synthesis method tends to be based on the phase of the precursor mixture before the resulted products, including solid state method, solution method, vapour method, and melt method [5].

Among all those synthesis methods, the solid state method has the simplest preparation but can be used to produce various product phase, including the single crystals [6], amorphous [7-10] and polycrystalline nanomaterials [11-16]. Some processes must be handled in high temperature [11,17,18], but in other case it can be conducted at low temperature, even at room temperature [19-23]. The synthesis tools include the conventional furnaces [16,24,25] and the modern ones such as microwave [25-29].

A review about solid state synthesis method was made but aboutprinciple of drug preparation and characterization method[30]. Another review studied three reaction types in the solid state methods including gas-solid, solid-liquid, and solid-solid reactions. No discussion about the detail synthesis steps and mechanisms[31].The solid state method was also reviewed but to study similarties and differences statistically to develop a predictive synthesis model[32].The solid state method for preparation of metal oxide nanostructure was reported but especially about thermal decomposition reaction of the metal-organic substance complexe molecules and macromolecules. The various examples were presented but no explanation about mechanism [33]. A review about synthesis of metal oxides was reported but about thermodynamics and synthesis route of the solid state method compared to three other methods (Pechini, combustion, and precipitation methods) which involved water solvent in preparation before calcination treatments[34]. Another one was about synthesis of transition and rareearth doped nickel spinel ferrite, but focused on comparation of the synthesis route between the solid state method and four others including hydrothermal, co precipitation, sol gel, and microemulsion[35].

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Received date 05 July 2025; Revised date 29 August 2025; Accepted date 30 August 2025

DOI: 10.21608/EJCHEM.2025.400966.12002

This paper presents more comprehensive review about solid state synthesis method than all those previous review papers [30-35], not only discuss the principle and steps, but also mechanism, advantage and disadvantages, influencing factors, the tools, and more various examples of the products such as salts, metal oxides, carbon, and composites.

2. Definition

There are threeimportant terms related to the solid state synthesis method including solid state synthesis (ceramic synthesis), solid state reaction, and ceramic method. Solid state synthesis or ceramic synthesis is a method which is used to change solid reactants to new substance with a definite structure by chemical reaction [36]. It is also defined as a method to produce inorganic material by burning the powder precursors at high temperature [37].

Ceramic method is a method which involve heating together two non volatile solids to react andproduce a product [38]. Another definition states that ceramic method isa synthesis method which involve grinding of powder mixture substances which contain metal (oxide, oxalate, carbonate) and heating the mixture at definite temperature, generally after pellet formation [39].

Solid state reactionis a method to make an inorganic solid by mixing powder reactant together and press them to form pellet or other shape before heating in the furnace [40]. Solid state reaction, also called as Shake"n bake reaction, is a asolid state method which the reactants are mixed and heated in the oven [41]. The third definition states that solid-state reaction refers to a conventional method used in chemistry to synthesize various materials like ceramics and crystals by heating a mixture of raw materials in solid form [42].

Based on those definitions, thosethree terms have same things including synthesis method, solid reactant,and heating process with each characteristics can be listed in Table 1.

Table 1:Comparison of methods based on their definitions

No.	Terms	Characteristics based on definitions	References
1.	Solid state synthesis (ceramic synthesis)	A method Reactant: solid, powder Process : chemical reaction, burning, high temperature Product: inorganic material with definite structure	[36, 37]
2.	Ceramic method	A method Reactant: non volatile solid, powder, pellet, metal oxide, oxalate, and carbonate Process: grinding of reactant, pellet formation, chemical reaction,heating process Product: no specific statement	[38,39]
3.	Solid state reaction (Shake'n bake reaction)	A method Reactant: powder , pellet or other shape Process: pressing of reactant, heating in the furnace Product: inorganic solid, ceramics, crystals	[40,41,42]

3. Steps and mechanism of crystal growth in solid state synthesis method

Solid state synthesis is classified in two main steps, including the starting and the crystal growth steps. The preliminer step includes weighing of reactant, grinding of precursor using agate and mortar, filling the grinded powder into the crucicle, pre heating for several hours, and heating. In the preheating step, the reactant decomposition and producesthe volatile gasses such as NH_3, NO_2, CO_2, dan H_2O and remain non volatile oxide only. The heating process is applied to homogenize the non volatil oxide mixture, to minimize the coarse solid particles, and to visualize the mixture as the germ. Germination starts when the phase is not in equilibrium. This condition is very affected by crucicle wall, impurities, and amorphous properties. Incrystal growth step, cations move to germ which form the layer. This migration prefers occur at high temperature. Growth of crystal occurs in several steps including arrangement of atoms, adsorption on the solid surface, diffusion, and fixation of atoms on the final sites. The sequent layers experience aggregation on crystal surface to increase the crystal volume **[**43**].**

The solid state reaction of MgO and Al_2O_3 which forms $MgAl_2O_4$ spinel can be used to explain crystal growth by solid state method. The shape, size, ideal arrangement, and ideal solid mixture are shown in Figure 1, although no uniform shape and regular arrangement in reality [2].

Nucleation lasts well by a heterogenous condition and similarity of two crystal reactant structures. In interface of $MgO/MgAl_2O_4$, the nucleous of $MgAl_2O_4$ and MgO are in the same crystallography orientation. Their arrangements don't change because both have the same ccp structure.There is a similarity of the MgO and spinel structures which make formation of spinel nucleous on top of MgO structure can last easily. In the MgO structure, the Mg^{2+} cations occupy all octahedral sites, on other wise in spinel structure, the Mg^{2+} cations stay in 1/8 tetrahedral sites with the Al^{3+} occupy ½ octahedral sites[40]. The Mg^{2+} ions in MgO and the Al^{3+} ions in Al_2O_3 are trapped on each their lattice sites. However, at very high temperatures, those ions have sufficient thermal energy to leave their sites [44]. The crystal structures of MgO, Al_2O_3 and $MgAl_2O_4$ are presented in Figure 2.

Wagner reaction mechanism explains the reaction mechanism of MgO and Al_2O_3 involving the Mg^{2+} and Al^{3+} diffusion through the product layer. This diffusion can be lasted because the ions have the sufficient thermal energy to leave their sites by heating treatment [44]. In the diffusion process, the Mg^{2+} and Al^{3+} ions move in different directions (Figure 3). The Mg^{2+} ions move away from the $MgO/MgAl_2O_4$ interface and go near to $Al_2O_3/MgAl_2O_4$ interface whereas the Al^{3+} cations move away and go near oppositely so that the layer of spinel grows up and thicker.

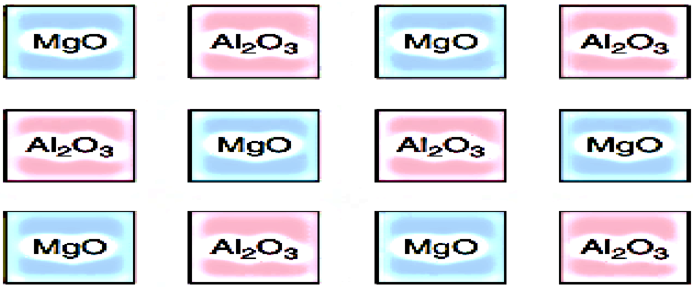

Fig. 1. Ideal mixture of MgO and Al_2O_3reactant solid grains [40].

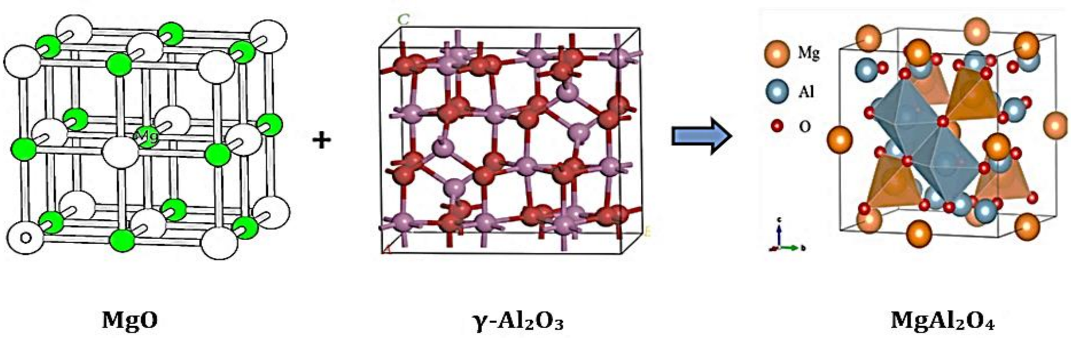

Fig. 2. Unit cell structures of MgO, Al_2O_3 and $MgAl_2O_4$ [45-47].

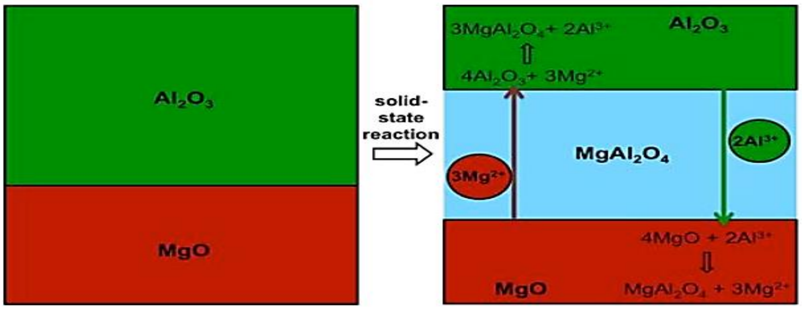

Fig. 3. Diffusion process of Mg^{2+} and Al^{3+} in interface of $MgO/MgAl_2O_4$ and $Al_2O_3/MgAl_2O_4$ interfacesin formation of spinel [48].

Two interface reactions are lasted including the reactions of MgO and $Al^{3+}$and the reaction of $Mg^{2+}$and $Al_2O_3$to form $MgAl_2O_4$ spinel. The rate-determining step for the reaction is the diffusion of Al^{3+} and Mg^{2+} cations through these interfaces [44]. The reactions in interface can be written as follows[40]:

Interface of $MgO/MgAl_2O_4$: $2Al^{3+} - 3Mg^{2+} + 4MgO \rightarrow MgAl_2O_4$
Interface of $MgAl_2O_4/Al_2O_3$: $3Mg^{2+} - 2Al^{3+} + 4Al_2O_3 \rightarrow 3MgAl_2O_4$
Total reaction : $4MgO + 4Al_2O_3 \rightarrow 4MgAl_2O_4$

The other imagination is given in Figure 4 [49] with reactions as follows:

At MgO side : $2Al^{3+} + 4MgO = MgAl_2O_4 + 3Mg^{2+}$
At Al_2O_3 side : $3Mg^{2+} + 4Al_2O_3 = 3MgAl_2O_4 + 2Al^{3+}$

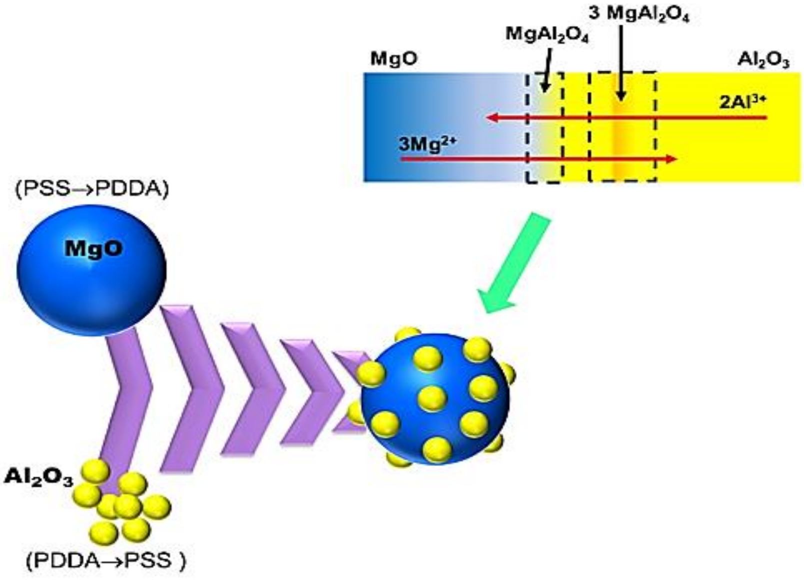

Fig. 4. Formation process of $MgAl_2O_4$ spinel by MgO and Al_2O_3 [49].

Characterization by STEM (Scanning Transmission Electron Microscope) to support the interface diffusion phenomenon between the Mg^{2+} and Al^{3+}cations in formation $MgAl_2O_4$ is given in Figure 5. In micrograph of STEM the yellow tetrahedral sites are the Mg^{2+} cations and the blue octahedral sites are the Al^{3+} cations in the spinel system.

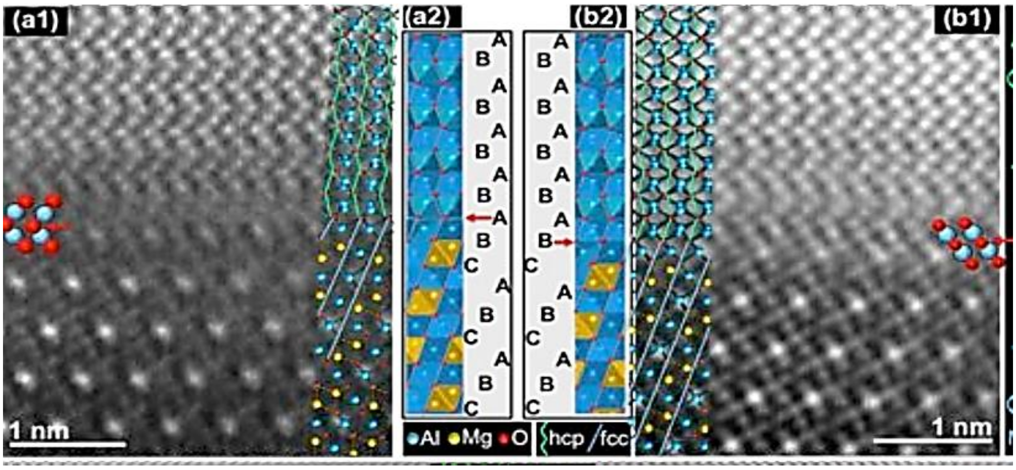

Fig. 5. STEM images which support interface diffusion in formation of $MgAl_2O_4$ spinel [48].

Atom diffusion is the migration of atoms among one lattice site to other ones. The atoms in solid materials move constantly and change position rapidly. There are two required conditionsincludingan empty adjacent site and sufficient energy of the atom to make the bonding with its adjacent atoms. There are some types of diffusions [50] as follows:

1. Vacancy diffusion : the interchange of an atom from a normal lattice position to an adjacent vacant lattice site or vacancy, as represented schematic alloy in Figure 6.

2. Interdiffusion or interstitial diffusion : it involves atoms that migrate from an interstitial position to an empty neighboring one. For example: interdiffusion of hydrogen, carbon, nitrogen, and oxygen, which have small atoms to fit into the interstitial positions.

3. Self diffusion : Diffusion occurs for pure metals, but all atoms exchanging positions are which have the same types.

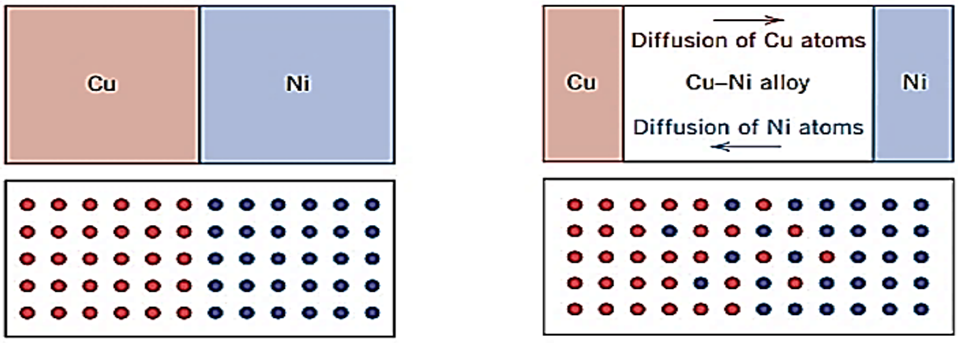

Fig. 6. Vacancy diffusion in formation of alloy CuNi [50].

The Wagner mechanism has 2 limitations: 1)Spinel existed as solid solutions at 1500°C with 50–60 mol% Al_2O_3 in the beginning of the reaction, but variable composition of the product, 2) the spinel product breaks away from the MgO parent during cooling due to the different volume of the phases which makes study of solid-state diffusion difficult [44].

4. Advantage and disadvantage of the solid state method

The solid state method is the simplest method and has relatively little parameters. However, those parameters are hard controlled. It can be applied to synthesize both single crystal [43,51] and polycrystalline [43]. The examples are listed in Table 2 and Table 3, respectively.

Table 2: Examples of single crystal synthesis by solid state method at low and high temperatures

No.	Products	Reactants	Synthesis conditions	Crucible	References
1.	$Ca_{1-2x}Zr_4M_{2x}P_{6-2x}O_{24}$ (M,Mo, x = 0.1 and 0.3)	$CaCO_3$, $(NH_4)_6Mo_7O_{24}.4H_2OZrO(NO_3)_2$ $(NH_4)H_2PO_4$	1250 °C for 72 h	Platinum crucible	[52]
2.	$0.98(K_{0.5}Na_{0.5})NbO_{3-0.02}(Bi_{0.5}Na_{0.5})(Zr_{0.85}Sn_{0.15})O_3$	K_2CO_3, Na_2CO_3, Nb_2O_5 SnO_2 Bi_2O_3	1150 °C for 15 h, 20 h, 30 h and 50 h with heating and cooling rates of 5 °C/min.	Double alumina crucibles with lids	[53]
3.	$Li_{1+x}[Ni_{0.6}Mn_{0.4}]_{1-x}O_2M$	LiOH, Li_2CO_3, Ni	925°C 20 h 950°C 12 h	Alumina crucibles	[54]
4.	Zn1-xLixO and Zn0.7Li0.28Mg0.02O	ZnO, Li_2CO_3 MgO	400 °C & 600 °C for 5 h each with a 5 °C per minute heating and cooling rates	-	[55]

Table 3: Examples of polycrystaline and nanomaterial synthesis by solid state method at high temperature heating

No.	Products	Reactants	Synthesis conditions	Crucible	References
1.	$Bi_{25}FeO_{39}$	Bi_2O_3, Fe_2O_3	650°C	-	[11]
2.	$BaBi_4Ti_4O_{15}$	$BaCO_3$, Bi_2O_3 and TiO_2	800 to 1100°C 24 h	-	[16]
3.	$BiFeO_3$	Bi_2O_3, Fe_2O_3	750 °C to 850 °C in air for 1 h	-	[17]
4.	$CsMnX_3$ (X = Cl, Br, and I)	Silica, zinc oxide Cs_2CO_3, $MnCO_3$, NaBr, NaI, NaCl, $PbBr_2$	1150°C 15 min	Corundum crucible	[24]
5.	$LiCoO_2$	$Co(OH)_2$	Sintered at 650 or 750°C	-	[57]

		$LiOH \cdot H_2O$, Li_2O_3	for 2 h in air		
6.	Nano SiC	SiO_2, Mg, C	600ºC	-	[58]
7.	$LaBO_3$	Lanthanum (III) oxide (La_2O_3) Boric acid (H_3BO_3)	500°C – 700°C 4 h	Ceramic crucible	[59]

The ceramic method tends to need the large process energy due to high process temperature (500–2000ºC). This is affected by the high coordination number in the ionic binner substances which need the high energy to overcome lattice energy and move by diffusion to the different sites. The coordination numbers of 4–12 are affected by charge and ion size. The unstabil substances which are decomposed at the high temperature can be handled by low temperature but slow reaction.The higher temperature the faster reaction and more increasing of diffusion velocity. Generally, the reaction temperature is lower than melting point so that it is called as solid state method [38].

By development, the solid state method can be performed by heating at low temperature (< 500 ºC), even without heating (at room temperature)as listed in Table 4 and Table 5, respectively.The solid state synthesis method at room temperature mechanically without heating is the green synthesis[60,61]. The solid state green synthesis has characteristics including environmentally friendly, solvent-free, less time, and low cost [62].

Table 4: **Examples of polycrystalline and nanomaterial synthesis by solid state method at low temperature heating**

No.	Products	Reactants	Synthesis conditions	References
1.	Cu_2ZnSnS_4 (CZTS) nanoparticle	$Cu(CH_3COO)_2.H_2O$) $Zn(CH_3COO)_2.2H_2O$ $SnCl_2.2H_2O$	250°C for 1 h in argon atmosphere	[65]
2.	VS_4	NH_4VO_3, thiourea	200ºC 5h	[66]
3.	Zeolite	$Na_2SiO_3.9H_2O$, fumed SiO_2, NH_4Cl, organic template	180ºC 24-48 h	[67]

The future benefit of the solid state synthesis method can be more improved by the combinatorial chemistry approach through self-driving laboratories by artificial intelligence, learning machine, and robot. Vision of this approach is to obtain fast data, saving time, and cost efficiency to provide marketable materials [63]. By using the rapid computational science and artificial intelligence technologies, prediction and high-performance solid state electrolyte can be calculated theoretically and emerged by AI method. AI methods have two strategies to build the relationships of the material structure and properties by the statistical algorithms and to perform the multi-layer neural networks for the complicated pattern recognition and extraction feature. These strategies provide a novel research for the designed and advanced materials [64].

Another development is usage microwave which can reduce the effective activation energy of the nucleation and growth in crystallization process. For example, the effective activation energy for the crystallization of PTO films by conventional heating and microwave irradiation was 216 kJ/mol and 131 kJ/mol, respectively [68]. The microwave can also reduce the electric energy consumption. When a muffle furnace needs the electric energy consumption of 4.39 kWh, a microwave oven can need only 0.3 kWh. For example, a muffle furnace (Nabertherm L 40/11) has an electrical power of up to 6000 W with a maximum temperature of 1100 ºC in a chamber of 320x490x250 mm. In other side, a microwave oven (Phoenix Black model with two magnetrons) has the lower maximum electrical power (3500 W, 2.45 GHz) with the maximum temperature up to 1200 ºC in larger room (400x730x305 mm) [69].

The solid state synthesis method has been applied in industries. For example the industrial grade cobalt aluminate pigment in Colorobbia Italia was synthesized by calcination at 1200 °C for 4 h [70].Another example is the $LiMn_2O_4$ powders synthesis by solid state method using the industrial microwave furnace with a multimode reactor system at 2.45GHz. In this process, the microwave irradiation was applied at temperatures to 400-1000°C for 5 and 30 minutes[71].

Requirement of solid state synthesis method is a chemical reaction in interface of 2 solids. When the surface layer has been reacted, more reaction will be lasted due to reactants can move in diffusion from bulk to interface. The interface reaction and diffusion through the solids will be faster in the high temperature. It is predicted that reaction time is 2/3 boiling point of the solid. However, diffusion is the limiting factor. Therefore, the precursor materials should have the small particle size and can be mixed well to maximize the contact of solid surfaces and to minimize space among reactants to make the diffusion. Reaction time tends to be in hour unite. For example, preparation of $CuFe_2O_4$ from Fe_2O_3 and CuO needs 23 h. The products are often inhomogenous because there is an oxide layer which is formed in reaction time at interfaces of the 2 crystals. It makes the ions need to move in diffusion through those layers before the reaction [38].

Table 5: **Examples of amorphous materials (glasses) and crystal synthesis by solid state green method without heating (at room temperature)**

No.	Products	Reactants	Synthesis conditions	Reaction container	References
1.	Ga_2S_3–Na_2S glass	Na_2S and Ga_2S_3	Ballmilling (ZrO_2 balls, 600 rpm)	Zirconia bowl	[7]
2.	ZrF_4–BaF_2 glass	ZrF_4 and BaF_2	Ballmilling (zirconia balls, 510 rpm)	Zirconia pots	[9]
3.	$NaGaS_2$ glass	Ga_2S_3 and Na_2S	Ballmilling (WC balls, 400 rpm	Tungsten carbide bowl	[10]
4.	Silver nanoparticles (Ag-NPs)	Dextran, NaOH, $AgNO_3$	Mechanical grinding	-	[15]
5.	Fe_3O_4@PANI nocomposites	Anilinehydrochloride, $FeCl_3.6H_2O$	Grinding in a mortar and hand-ground, 50 min	Mortar	[60]
6.	Silver nanoparticles (AgNPs)	$AgNO_3$, Thymus vulgaris L., Sambucus nigra L. and Thymus serpyllum L. plants	Planetary ball mill, 15 tungsten carbide) milling balls (10 mm diameter), ball-to-powder ratio 37, milling speed 500 rpm and milling time 2.0 h.	-	[61]
7.	$La_2(C_4H_4O_6)_3$ nanocrystallite	$LaCl_3·7H_2O$, $La(NO_3)_3·6H_2O$, $La_2(SO_4)_3·9H_2O$ $La(Ac)_3·3H_2O$, $Na_2C_4H_4O_6·2H_2O$	Grinding with infrared light irradiation	Agate pot	[73]

The way to make easier reaction by solid state method is usage of reactive reactant or the reactant which has the diffusing ions. The other problem is the volatile reactant (alkali metal oxides, PbO, HgO, Tl_2O, Bi_2O_3) or reactive to the reaction place. All those problems can be overcomed by adjusting the right synthesis procedure [40].The reagents should have high purity, because some impurities can give the undesired phase and affect the material properties. Beside that, this method can lead to the formation of the heterogenous powder with large solid particles. Therefore, this method requires the high temperature reactions for hours even days[73].

Synthesis of spinel by solid state reaction has a problem due to inert reactants such as MgO and Al_2O_3. Inert precursor means that the precursors are stabil and unreactive. The spinel formation reaction is slow due to very slow diffusion of the Al^{3+} and Mg^{2+}cations. The pure spinel can be taken at 1500ºC for 1 week. Reduction of reactant and product amount are needed to create the empty sites to make the ions can jump into the sites. The high temperature is needed to make the ions which have enough energy can go out from one site to the others [40]. There are reagents which are hygroscopic and highly sensitive to water vapor. For example, lanthanum oxide (La_2O_3), reacts with air to form carbonates and hydroxide quickly, therefore it should be calcined first at 900 °C to remove all those impurities [73].

The solid state reaction is hard if the reactants are separated. The way to make faster reaction is by grinding of the mixture in half reaction to destroy reactant and product interface and to make contact of the reactant surfaces. The other way is by adding the transporting agent which has liquid phase or gas to bring reactants together without long solid phase diffusion time. Addition of the little transporting agent (gas or liquid) is effective to improve the solid state reaction. No simple way to monitor a solid state reaction development. The trial and error waysare usually applied to monitor the reaction condition by using XRD or the other methods. This hard way makes people often mix the reactant and the product. Generally it is difficult to separate the product and the reactant even if the reaction is almost completed [40].

5. Influencing factors in the solid state method

There are some important things which must be cared in the synthesis using the solid phase method, as discussed in this section.

5.1. Choice of reactant material

The reactant materials must be stoichiometrically acurate, reactive, and relatively pure because they will affect quality of the product. For synthesis of SiC, purities of the products are different due to different silica precursors with sequence of SiO_2 (fumed) > SiO_2 (pp) > SiO_2 (sand) > SiO_2 (rice husk) with purity sequence of 99.8 % > 99.6% > 98.5% > 90.0%, respectively[74]. The chemical reaction as follows :

$$2SiO_2\,(s) + C\,(s) + 4Mg\,(s) \rightleftharpoons Si(s) + SiC\,(s) + 4MgO\,(s)$$

Formation of SiC is also affected by the reactant composition. For the Na and S mass ratio of 4:2.4 and the S and Si mol ratio of 3:1, the SiC substance can be successfully formed at 300°C. However, when those ratios were changed to 2:0.8 and 1:1, respectively, the product was amorphous [75]. The chemical reaction as follows :

$$Si\ (s) + C\ (s) + 2nNa\ (s) + nS\ (s) \rightleftarrows SiC\ (s) + nNa_2S\ (s)$$

In synthesis of oxides, the acetate, carbonate and nitrate salts can be used as the reactants because they are decomposed in initiation step by heating process. In the decomposition step, the particle size decreases and the surface area increases so that the potential reactivity is improved. Mixing the solid phase reactants can produce the gas evolution along decomposition process [40]. Examples of the decomposition reactionsfor the salts are listed in Table 6.

The impurities in the salt reactants can affect the decomposition reaction effectivity and the temperature of decomposition reaction. For example, by using TGA analysis it can be confirmed that the solar salt (60 wt% $NaNO_3$, 40 wt% KNO_3) was stable in heating in the air until 500oC and experienced the thermal decomposition at the range of 500 – 900 (steep weight reduction) and 900 -1000 °C (ramp weight reduction), indicating the thermal decomposition of the nitrate to nitrite and of the nitrite to the metal oxide. In other side, $Mg(NO_3)_2.6H_20$ showed three ranges of decomposition reaction temperatures, including 50-150°C, 190-320 °C, and 350-450 °C which are probably related to dehydration reaction, the decomposition reaction from salt nitrate to nitrite, and from nitrite to metal oxide, respectively. The chemical reactions for basic thermal decomposition reaction of the metal nitrates as follow [76]:

$$MNO_3\,(s) \xrightarrow{\Delta} MNO_2\,(s)\ \ 0.5O_2\,(g)$$

$$2MNO_2\ (s) \xrightarrow{\Delta} M_2O\ (s) + NO_2\ (g)\ + NO\ (g)$$

Substance types, especially different anion types of the salt reactant (Cl^-, NO_3^-, SO_4^{2-}, CH_3COO^-) can affect % yield and properties of the product such as particle size and morphology as listed in Table 7. Among those all salt types, the $La(Ac)_3.3H_2O$ produced the smallest particle size and the largest % yield.

Table 6: Examples of thermal decomposition reactions

No.	Reactant	Reaction equation / product	Condition	References
1.	$BaCO_3$	$BaCO_3 \rightarrow BaO + CO_2$	673K 24 h	[77]
2.	$CaCO_3$	$CaCO_3 \rightarrow CaO + CO_2$	-	[78]
3.	$ZnCO_3$	$ZnCO_3 \rightarrow ZnO + CO_2$	-	[79]
4.	$Co(CH_3COO)_2.\ 4H_2O$	$Co(CH_3COO)_2.4H_2O \rightarrow Co(CH_3COO)_2 + 4H_2O$ $Co(CH_3COO)_2 \rightarrow CaCO_3 + (CH_3)_2CO$ $CoCO_3 \rightarrow CoO + CO_2$	40-120°C 180-260°C 263-300°C	[80]
6.	$Zn(CH_3COO)_2.2H_2O$	$Zn(CH_3COO)_2.2H_2O \rightarrow Zn(CH_3COO)_2 + 2H_2O$ $Zn(CH_3COO)_2 \rightarrow Zn + (CH_3)_2CO + CO_2 + ½\ O_2$	55-100°C 160-290°C	[80]
7.	$Hg(CH_3COO)_2$	$Hg(CH_3COO)_2 \rightarrow Hg + (CH_3)_2CO + CO_2 + ½\ O_2(g)$	1600-300°C	[80]
8.	$Pb(CH_3COO)_2.2H_2O$	$Pb(CH_3COO)_2.4H_2O \rightarrow Pb(CH_3COO)_2 + 4H_2O$ $Pb(CH_3COO)_2 \rightarrow PbCO_3 + (CH_3)_2CO$ $PbCO_3 \rightarrow PbO + CO_2$	40-200°C 240-300°C 310-370°C	[80]
9	$Mg(CH_3COOH)_2$	$Mg(CH_3COOH)_2 \rightarrow MgO + CO_2 + H_2O$	600°C with N_2 as protective gas	[81]
10.	$Cu(CH_3COOH)_2$	$Cu(CH_3COO)_2.H_2O \rightarrow Cu(CH_3COO)_2 + H_2O$ $Cu(CH_3COO)_2 \rightarrow Cu + 2CH_3COO^-$	111-159°C 208-317°C	[82]
11.	$Co(NO_3)_2.xH_2O\ /Al_2O_3$	Co_3O_4, $CoAl_2O_4$, Al_2O_3	350°C	[83]
12.	$Ni(NO_3)_2.xH_2O/Al_2O_3$	NiO, Al_2O_3	400°C	[83]
13.	$Mn(NO_3)_2.xH_2O/Al_2O_3$	Mn_2O_3, Al_2O_3	600°C	[83]
14.	$Zn(NO_3)_2.xH_2O/Al_2O_3$	ZnO, Al_2O_3	350°C	[83]
15.	$Cu(NO_3)_2.xH_2O/Al_2O_3$	CuO, Al_2O_3	350°C	[83]

Table 7: Yield and properties of f nanocrystalline $La_2(C_4H_4O_6)_3$ under different reactant materials by solid-state synthesis

No.	Reactant materials	Synthesis conditions	Morphology	Particle size (nm)	Yield (%)
1.	$LaCl_3.7H_2O$, $Na_2C_4H_4O_6$	Grinding under infrared light, 35 -40°C, ultrasonic, 30 min	Approx ball shape	80	89.1
2.	$La(NO_3)_3.6H_2O$, $Na_2C_4H_4O_6$	Grinding under infrared light, 35 -40°C, ultrasonic, 30 min	Approx ball shape	90	90.2
3.	$La_2(SO_4)_3.9H_2O$, $Na_2C_4H_4O_6$	Grinding under infrared light, 35 -40°C, ultrasonic, 30 min	Rhombus shape	70	90.2
4.	$La(Ac)_3.3H_2O$, $Na_2C_4H_4O_6$	Grinding under infrared light, 35 -40°C, ultrasonic, 30 min	Rhombus shape	60	92.6

Source: [72]

5.2. Mixing technique of reactants

Good contact of the reactant particles by increasing the surface area of reactants is the important key in solid phase reaction. The manual technique is mixing and grinding using agate and mortar. The other technique is by mechanic machine such as ball mill. The principle of ball mill is by putting the reactant mixture in the rotating container with some agate balls [40]. In ball mill the container rotates for definite period with the balls inside to reduce the solid particle size to get the homogenous reactants.

The higher energy of ball mill the faster and more effective the mixing process. It is possible to add the liquid for milling, such as water or organic liquid [38]. For example, $PtCl_4^-$ sodium hexafluoroacetylacetonate reaction mixture after grinding 4 h and 3 h gave different crystallinity of the product [84]. The mechanical milling of reactants ($BaCO_3$, $SrCO_3$, and TiO_2) for 20 h resulted in their higher crystallinities than for 60 h due to crystal defect [85].

5.3. Reaction container (crucible)

If contacting with atmosphere is not the problem, the reactant mixture can be heated in furnace with air using the suitable crucicle. The reaction container must be inert toward the reactant and stable at the high temperature. The most used crucible are silica (1157 °C), alumina (1927 °C), zircon (2027 °C), or magnesia (2427 °C). Tantalum, platinum, and graphite are also used as the containers [38]. The examples of the containers and their characteristics are listed in Table 8. Alumina crucibles have cheaper price than platinum crucibles. However, alumina crucibles tend to contaminate the melts, which affects the glass product properties [86]. For example, changes of sodium phosphate glasses thermal properties have been investigated for using Pt/Au VS fused alumina crucibles. Glasses were produced from NaH_2PO_4 as a reactant material. In Pt/Au crucibles, glass transition temperatures rose to 295°C. In fused alumina crucibles, glass transition temperatures rose to over 450°C. Extensive erosion of the alumina crucibles was observed [87]. $Bi_2Sr_2Ca1Cu_20_y$ glasses which were processed using alumina crucible contained 2.26 % of Al, but if using Pt crucible it contained 0.02% of Pt [88].

Although worse crucible for production of glasses than Pt/Au crucibles, the alumina crucible is better than the lower cost ceramics such as cordierite or fireclay due to its higher level of corrosion resistance in high temperature[89].Addition of Al_2O_3 to Pt as Pt-Al_2O_3 composite as the crucible also can improve strength of the crucible from 155 to 235 MPa at 20°C [90].

Table 8: Crucibles and their characteristics

No.	Cruclible	Characteristics
1.	Pt	Pt crucible can be applied to 1200°C, strong in high temperature, chemically inert, resistant to oxidation and corrosion by acid and salt melt. It is reactive toward BaO, Li_2O, and transition metal oxides.
2.	Au	Au has boiling point of 1063 °C but more inert than Pt.
3.	Al_2O_3	The highly pure alumina is inert and has high boiling point. However, the reactant can contaminate it.
4.	SiO_2	Glass SiO_2 crucible can be used for reaction at more than 1200°C, but it can oxidize alkali metal.
5.	Graphite	Graphite crucible is often used for synthesis of sulfide, nitride, and calchogenide.
6.	Porcelain	Porcelain (mullite crucibles) can be used to 1050°C, cheap, but reactivetoward HF and alkali salt melt.
7.	Teflon	Teflon or polytetrafluoroetylene (PTFE) is inert and can be used from -196 to 280°C.
8.	Stainless steel	Stainless steel crucible can be used in the range of from –180 to 800°C, resistant to concentrate alkali, nitrous acid, nitric acid, and kalium permanganate, acetic acid, boric acid, organic acids, and solutions of sulphate, phosphate, and bromide.
9.	Nikel	Ni crucible 99.9% has impurities of Ti and Mg, hard to be oxidized in the air, can be used to 600°C, resistant to NaOH, Na_2O, alkali, and sea water, chlorine, HCl gas, HCl, H_2SO_4 and HNO_3.
10.	Zr	Zr crucicle can be used to 450°C, resistant to Na_2CO_3 atau Na_2O_2, alkalin mixture, carbonate, hydroxide, peroxide, borate, nitrate, and fluorite.
11.	Steel	Steel crucible can be used to 500°C.

Source : [91]

5.4. *Heating adjustment*

Heating process is designed to : a) decompose slowly metal oxides reactants without foam and too much leakge, b) prevent volatilization of one or more reactants, c) use the right temperature for definite period and definite reagent [40]. For example, a layered structure $LiNiO_2$ was synthesized by the solid-state reaction under air atmosphere in range of 750 °C to 850 °C for 20 h using Li_2CO_3 and NiO as precursors. XRD characterization showed that the higher calcination temperature the smaller crystal size, the smller unit sel , and the lower crystallinity based on intensity of X-ray diffractograms [92].

The solid phase reaction can achieve 2027 °C so that it needs the furnace which is resistant to the heating. Laser carbondioxide (with output of infrared) can give temperature of 4027 °C. The atmosphere can be controlled if we use tube furnace. The other alternative is usage of silica glass or noble metal to prevent volatile reagent evaporation [40].

The synthesis by using microwave can reduce reaction time and temperature becausethe reactant components can absorp the microwave radiation which increase both reaction velocity and diffusion velocity. The advantage of microwave usage is the heating processstarts from the inner of the reactants and spread to all parts of them fastly with efficient energy. It is different from the conventional furnace because microwave has 3 characteristics of heating including direct heating, volumetric heating, and instantaneous heating [93]. The examples of the microwave and synthesis of metal oxides using microwave are presented in Figure 7 and Table 9, respectively.

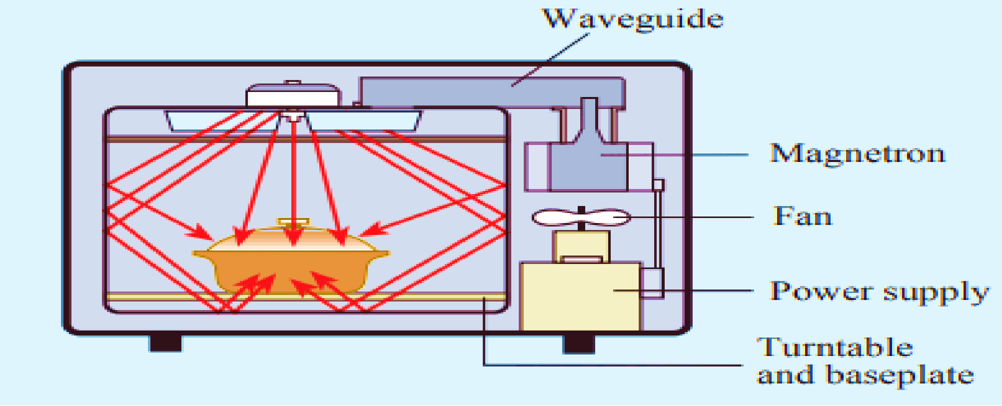

Fig. 7. Microwave furnace model [94].

Table 9: Comparation of microwave and conventional solid state method

No.	Products	Synthesis conditions		Product properties		Ref
		CV	MW	CV	MW	
1.	$MgAl_2O_4$	550 °C, 6 h	700 W, 60min	Crystalinity of 44%; particle size of 7.99 µm, S_{BET} of 8.14 m^2/g	Crystalinity of 66%; particle size of 5.43 µm, S_{BET} of 36.03m^2/g	[25]
2.	$ZnAl_2O_4$	1400 °C	1200 W	Crystalinity of 93.5%	Crystalinity of 95.0%	[26]
3.	Al_2O_3/SiC ceramic	1500 °C, 300 min	0.9 to 2.4 kW, 15min	Relative density of 98.2 %; Average matrix grain size of ~4.7 (µm)	Relative density of 99.7%; Average matrix grain size of ~3.6 (µm)	[29]

6. Synthesis of carbon and composites with solid state methods

In this section we especially present review about our own researchs including synthesis of CNM or CNS, modification of kaoline, and synthesis of composite such as kaoline–CNS composite and spinel–CNM composite.Biomass or bioresource is all organic substances derived from green plants after processes of digestion, burning, or decomposition.The carboneous materials were synthesized using the various biomass by pyrolysis reaction. The biomass contain thelignocellulosic substances which are the carbon precursors including cellulose (40-60%), hemicellulose (10-30%), and lignin (20-30%). In pyrolysis process, the lignocellulosic substances experience the thermal decomposition reactions [95].The other biomass such as various flours contain complexe carbohydrates such as amylose and amylopectin [96].The organic substances in the biomass which were used in our researchs are listed in Table 10 and Table 11.

Table 10: Contents of lignocellulosic substances in some plant biomass

No.	Flour	Amylose (%)	Amylopectin (%)
1.	Tapioca	16.7	83.3
2.	Corn starch	28	72
3.	Wheat flour	20	80

Source: [96]

Table 11: Contents of lignocellulosic substances in some plant biomass

No.	Biomass	Cellulose (%)	Lignin (%)	Hemicellulose (%)	References
1.	Coffee cherry	27.6	13.7	12.5	[95]
2.	Green Tea	14.26	52.44	33.33	[95]
3.	Patchouli	42.12	24.45	18.72	[97]
4.	Rice husk	32.67	17.01	22.82	[97]
5.	Sugarcane leaves	22.65	34.24	17.19	[97]
6.	Coconut fiber	18.48	36.29	20.23	[97]
7.	Sawdust	22.06	50.30	14.95	[97]

Substitution of kaoline using various transition metal salt chlorides (Figure 8) was conducted with dry solid state reaction at 1100° C for 5 h using a conventional furface [98]. Changing of kaoline color and shift of FTIR spectra wavelength, especially at 738.69 cm^{-1} (Si-O-M) and 813.90 cm^{-1} (M-O) indicate substitution of Al^{3+} by metal cation (Zn^{2+} and Fe(III)) on framework of kaoline. XRD characterization indicated the intercalation of hematite and maghemite in the space between kaoline layer [99].

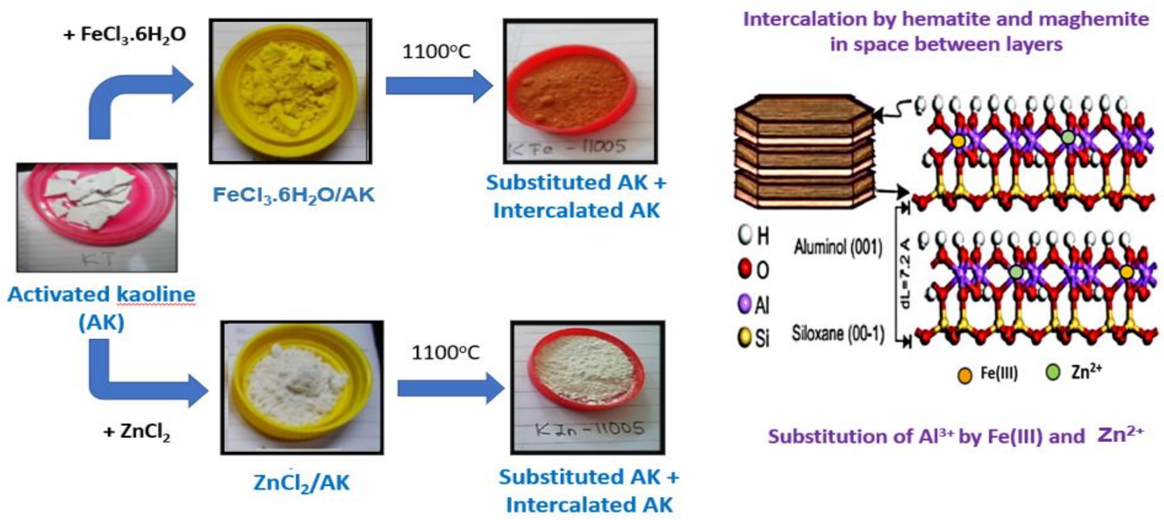

Fig. 8. Substitution and intercalation of kaoline using solid state method [99, 100].

Synthesis of CNS was performed using precursors of various expired flours (tapioca, maizena, rengginang, wheat flour, polard) and expired drinks (green tea, coffee powder) and a chemical activator ($ZnCl_2$) with dry microwave method at 800 W for 50 minutes, followed dispersion of the activated carbon in the water by using mixer for 1 h (Figure 9). Among all precursors, the coffee based CNS had the highest colloid concentration (the highest TDS). The mixer was better for dispersion process because it gave higher colloid concentration. Based on stability test, the tea based CNS had more stable colloid than the others [101].

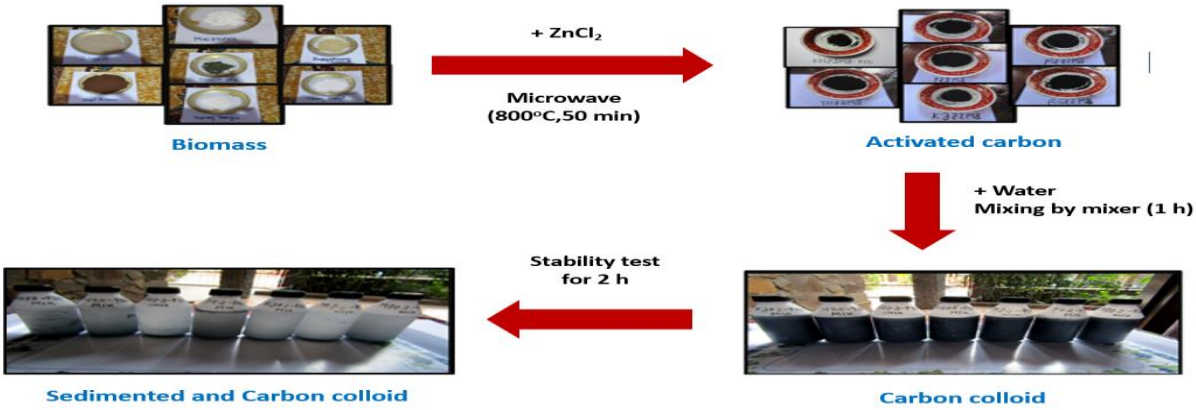

Fig. 9. **Synthesis of CNS from various expired flours and drink using solid state method [101].**

CNM was synthesized by pyrolysis of various biomass (patchouli, rice husk, sugarcane leaves) by sequent hydrothermal and microwave method (Figure 10). The composite of MFe_2O_4/CNM (M = Zn, Ni, Mn) were synthesized from mixture of CNM, salt chlorides, and KOH using the conventional furnace at 600°C for 15 minutes. Composites of rice husk based $ZnFe_2O_4$/CNM gave the highest removal of pesticide by degradation in the dark [102-106].

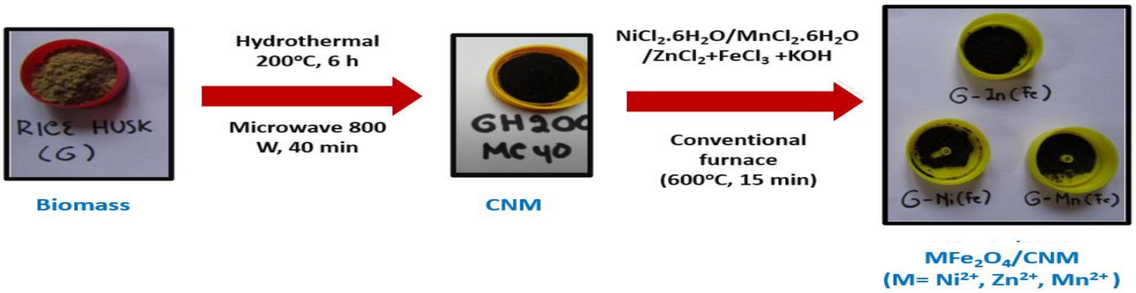

Fig. 10. Synthesis of MFe_2O_4/CNM composite by using solid state method [102-106].

Various biomass (patchouli, sugarcane leaves, rice husk, coconut fiber, and sawdust) were pyrolized with dry microwave method using chemical activator of $ZnCl_2$ in the closed porcelain crucible at 400, 600, and 800 W for 50 minutes (Figure 11). The activated carbon products were dispersed in the water and blended using blender. The nanocarbons were mixed with $ZnCl_2$, $CrCl_3$ and KOH and wetted with little water then calcined using microwave at 600W for 5 minutes. The activated carbon and the composite of $ZnCr_2O_4$/AC were verified using XRD characterization. Test of pesticide degradation in the dark showed that the sawdust based $ZnCr_2O_4$/CNS composite gave the highest TOC [97,107]. Two of all biomass are presented in Figure 11.

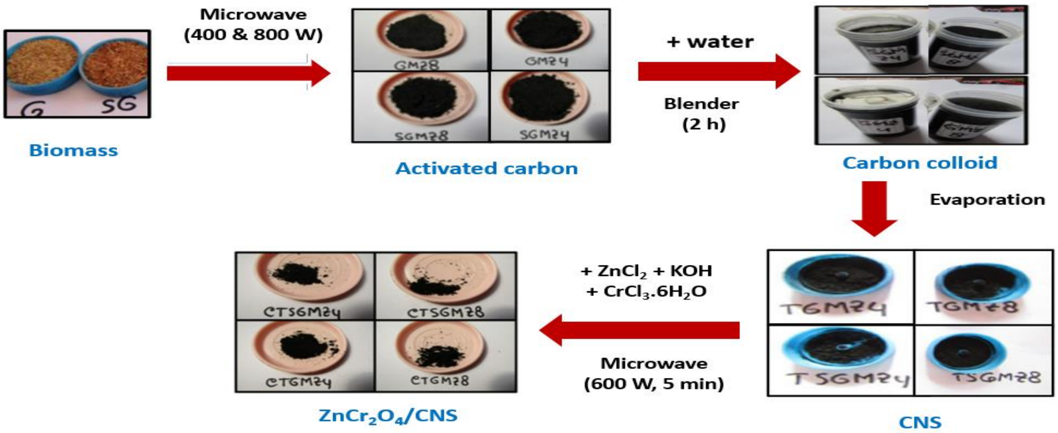

Fig. 11. **Synthesis of CNSs and their composites from rice husk and sawdust using dry microwave [97,107].**

Introduction study was conducted about effect of container shape toward carbonization reaction of coffee. The coffee with brand of Kapal Api Spesial (KAS) was used as the precursor. There were two kinds of containers, including earthenware cup (CG) and kettle (CR). Pyrolysis process for the introduction research is presented in Figure 12. Along pyrolysis, the CG cover was opened slightly to reduce pressure inside CG by removing the volatile gasses which were emitted by pyrolysis reaction.

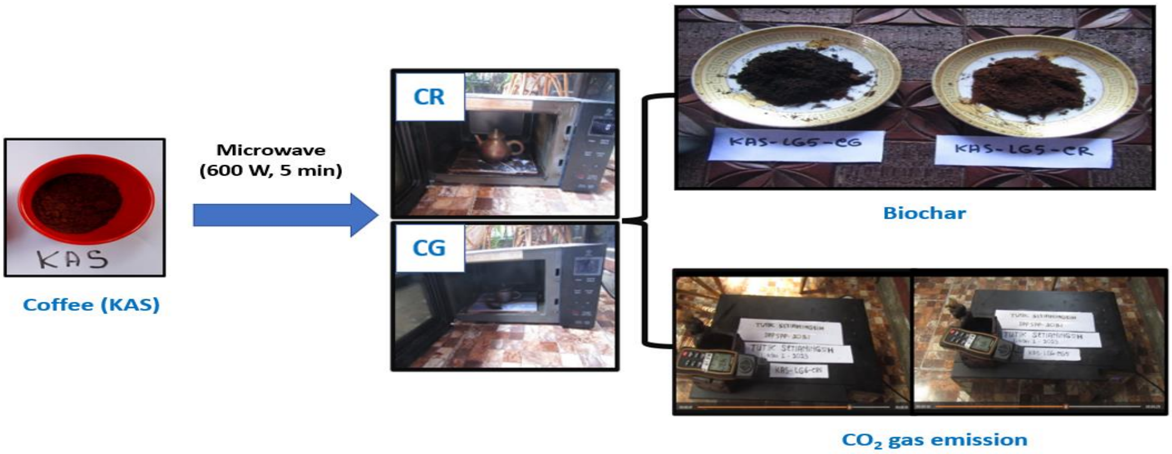

Figure 12. **Pyrolysis of coffee (KAS) using containers of earthen kettle and cup by solid state method [108,109].**

Figure 12 confirms that the carbon, which was produced by using CG, tends to be blacker than using CR. That is probably smaller room so that CO_2 gas is more optimal to make contact again with surface of the carbon and activate it. Beside CO_2, the water vapour which was emitted also can activate the carbon:

$$C(s) + CO_2\,(g) \rightleftharpoons 2CO\,(g)$$
$$C(s) + H_2O\,(g) \rightleftharpoons CO\,(g) + H_2\,(g)$$

Figure 12 shows difference of the emitted carbon dioxide from the microwave along pyrolysis process. Pyrolysis in the earthen kettle (CR) emitted 430 mg/Kg CO_2 gas, whereas, earthen cup (CG) , emitted 447 mg/Kg. This difference probably is caused or room of CR is larger and need long time to go out from the container than CG.

The other study for introduction research (Figure 13) was performed about effect of activated kaoline (KT) and the precursor ratio toward mass of koloid composite and sedimented composite after pyrolysis using solid state method [110,111]. The precursors were tea (TH) and coffee (KAS) and the ratio of KT and TH or KAS were 3:1 (code: KT3KAS1 and KT3TH1) and 1:3 (KT1TH3 and KT1 KAS3). In synthesis process, activated kaoline, tea/coffee, $FeCl_3$, $ZnCl_2$, and KOH mixture was calcined at 600 W for 5 min. The products were dispersed in water using mixer for 2 h to get colloid because the colloid has dispersed particle size of 1-100 nm [112]. The nanomaterial size is 1-100 nm [113,114].

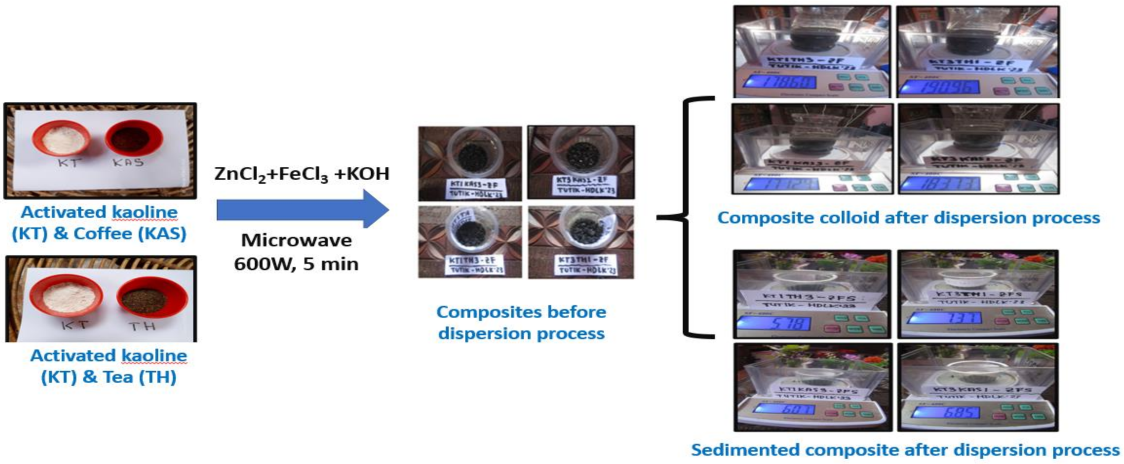

Fig. 13. Synthesis of modified kaoline – carbon composite using solid state method[110,111].

Figure 13 confirms that more activated kaoline amount made larger mass of colloid. It is possibly due to more polar kaoline surface than the carbon one. Kaoline is rich of -OH on the surface which makes stronger to make interaction with water solvent using hydrogen bonding. For the same ratio of activated kaoline and precursor, usage of tea tends to make larger mass of the sedimented composites. This difference probably is because aromatics structure in components of tea is more than in coffee.Brand of KAS is the roasted robusta coffee. Its main contents are carbohydrate, fat, and melanoid. The main carbohydrate in the coffee is polysacaride [115]. The green tea has main contents of catechin, fiber, and protein [116]. Coffee and tea also contain the different lignocellulose composition. The spent coffee ground contains 31.9 % lignin, 49.8% cellulose, and 15.4% hemicellulose. In other side, the green tea contains 52.44%; 14.26%; 33.33%, respectively [117].

7. Conclusion

Principally, a solid state synthesis method is a dry synthesis or by wetting with little solvent at high temperature (500-2000°C). The advantages of the solid state method are the simple preparation, single crystal, polycrystaline products, glass and it can be set at low temperature (< 500°C) even at room temperature. The disadvantages of this method included the requirement of the reactive and unvolatile reactants, relatively long synthesis, and hard monitor. The microwave assisted solid state synthesis overcomed the long time problem and the mechanical solid state method at room temperature provided the lowest cost, solvent-free, and environmentally friendly. However, more improvement for future is still needed by the combinatorial chemistry approach through theoretical calculation, artificial intelligence, learning machine, and robot.

The steps of synthesis are the preliminer and crystal growth. The mechanism of synthesis are diffusion (vacancy diffusion, interface diffusion, and self diffusion). The influencing factors of this method are reactant choice, container type, heating adjustment, and reactant mixing technique. The important tools for synthesis with this method are the mortar, pastle, hydrolic pressure, tube furnace, conventional furnace, and microwave. The solid state method has been applied for synthesis of metal oxides, silicon carbide, metal salts, zeolite, modification of kaoline, synthesis of CNS, CNS-spinel composite, and CNS-kaoline composite. All those resumes can be presented in a graphical abstract in Figure 14.

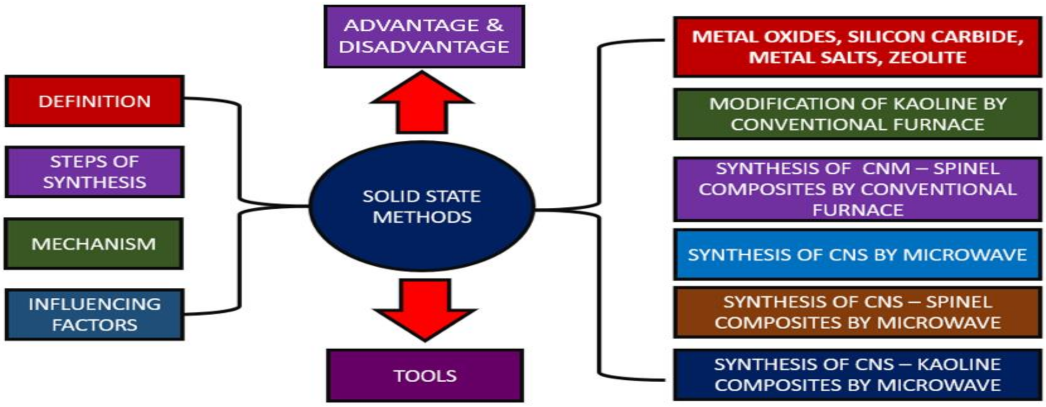

Fig.14. Solid state synthesis method : Principle and applications.

8. Conflicts of interest

"There are no conflicts to declare".

9. References

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