## MECHANISME OF FLOW INJECTION SYNTHESSIS STAGE IN HALF FRACTION THE REACTION CuZnFerrite

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#### Abstract

Mechanisme of Flow Injection Synthesis stage in half fraction of the reaction CuZnFerrite have been done in 2012, where the material electricity still existence untill now. There are additional accessories that merge into one namely; Sprayers, reagents, hoses and peristaltic movers. The four accessories work using a certain amount of energy but have no effect on the process. Flow Injection Synthesis process occurs after the accessories work. But its existence must be considered, especially half step. The reaction product is collected in a container, cleaned and diluted so that the pH of the solution is = 7, Then it is precipitated and dried

*Keywords:* Reliability, reliable, valid, Avrami, Ozawa, Kissinger, cooling rate, Flow Injection Synthesis, diversivication

Prior to testing stoichiometric calculations need to be done. This is done to determine the behavior of elements of chemical elements that must occur. Usually that should not happen is a material that is difficult to form and inconsistent physical form material. Things that may occur include continuous changes from a liquid to a solid yield, but do not directly clog the capilery pipes.

There are 4 compounds of salt that have a solid background of the salt of Cu, Zn Fe2 and Fe3. The four compounds allow to solidify. In the Flow Injection Synthesis reaction co-precipitation of yields formed in the form of solids that precipitate and sediment water will be separated aloud.

The reaction to Flow injection synthesis is a complete reaction. After the reactant reaction process direator and skipped capillaries for complete conditioning. In the capillary pipe is expected to occur a perfect reaction between acid and base. Out the pipe is no reaction, and if precipitation occurs precipitation is actually a precipitate material.

The total amount of the reactant solution was reacted precise volume depleted. Referring to the solution that must flow, the reactants will be yield (precursor) is certainly smaller volume compared to the alkaline reactant follows. As an example of CuZnFerrite material, if the material is reacted correctly following the stoichiometry then the precipitated material is the reacting material. If excess acid or an excess base, excess acid or base will form liquid.

- 1. Stoichiometric produce all forms mole ratio of reactants and all yield. In the coprecipitation reaction is required reactants in the form of saturated and even supersaturated solution.
- 2. 2 FeCl2.4H2O + 4 FeCl3.6H2O + 3x CuCl2.2H2O + 3 (1-x) ZnCl2 + 22 NaOH ->
- 3. 3 CuxZn (1-x) Fe3 04 + NaCl + 22 (42 + 6x) H20 .....1

Precurso	r Solution				Reagent so	lution.	
Fraksi CuCl2,in dex 2x	Fraks ZnCl2,index 2(1-x)	FeCl2 4H2O	FeC13.6H20	Total Volume Prekurs or	Solution of NaOH	Real Solution pH=13.7	Stochio metri
3x[gram /20ml]	3(1- x)gr/20ml	[gram/20 ml]	gram/20ml	[ml]	[gram/20 ml]	[gram/800 ml	
0	4.089	7.9532	21.6264	33.668 6	17.6	17.6	ZnFe <sub>3</sub> O <sub>4</sub>
2.04456	3.2712	7.9532	21.6264	34.895 36	17.6	17.6	Cu <sub>0.2</sub> Zn <sub>0.8</sub> Fe <sub>3</sub> O
4.08912	2.4534	7.9532	21.6264	36.122 12	17.6	17.6	Cu <sub>0.4</sub> Zn <sub>0.6</sub> Fe <sub>3</sub> O
6.13368	1.6356	7.9532	21.6264	37.348 88	17.6	17.6	Cu <sub>0.6</sub> Zn <sub>0.4</sub> Fe <sub>3</sub> O <sub>4</sub>

The reactor is an injector, which has two inputs, a special input for the acid and a second input specially for alkali. With a predetermined comparison the two materials are reacted. As an example, see the schematic drawing solution channel entrance reactor.

Batch shape injector is described in more detail as follows,



Figure 1. Schematic Diagram Injector Process.

The Injector parts diagram, conditioner laminar flow yield

Flow Injection Ferrite Materials Synthesis.section essential part consists of;\

- 1) inlet for saline.
- 2) The inlet for alkaline solutions.
- 3) The nozzle channel salt solution.
- 4) Space Reactor (Bath reactor)
- 5) laminar flow conditioning channel (channel orifice).

6) Hose conditioning particle growth.

In order to obtain complete reaction between the salt solution as a precursor to the super-saturated solution of lye, which need to be considered, among others;

o Availability of the two reactants sufficiently.

o When interfacing the two reactants in the reactor bath enough.

The mechanism of the process described as follows; Nucleation mechanism at the solid-liquid system is divided into two categories, namely:1. Primary Nucleation (primary nucleation). Nucleation due to the incorporation of molecules of dissolved substance (solute) to form clusters, which later grew into In a supersaturated solution (supersaturation), there will be the addition of solute crvstals. that diffuses into clusters and grow to be more stable. Crystals enlarged size, solubility caused down, otherwise if the crystal size is small then the solubility rose. Therefore, if there is a larger crystals then

crystals will grow, while the smaller crystals will be dissolved again, this is called the Theory of Miers. Seen: cooling solution (having the condition in point a).During cooling to pass solubility curve has not crystallize. Cooling continues until the point b, and the crystals begin to form, and the concentration of the solution be at point C (as the mother liquor / mother liquor).Solubility curve is the boundary where the nucleus formation begins spontaneous, and crystallization began to form. There is a trend: the super solubility curve as an area where the nucleation rate increased sharply.

2. Flow Producing Nozzel Secondary Nucleation (Secondary nucleation), Nucleation occurs when crystals collide with other materials, mixer, wall / pipe tank. Nucleation can be accelerated by the presence of seed crystals, the activation energy is smaller than the primary nucleation. Adder seed-seeding. crystals (small) at the beginning of the synthesis. For the purpose of the primary and secondary nucleation then the process is done in one reactant flow is laminar but have the high particle collisions, these properties occur in laminar flow that passes through a small gap or orifice or flow out of the nozzle, Primary and secondary nukleason in batch injector can be predicted from a position within the batch solution.

The shape of the particles in a batch reactor zone can be estimated by tracing percentage yield formation in the span of a solution of the reactants and yield journey across the zone.

At the time of the precursor solution moves out nozzle, particle precursor particles and the leading edge portion of the solution will intersect with an alkaline reactant particles will be formed nucleation followed by growth Krista yield particles. The motion of particles now start getting complex. At first move in the direction of the axis of the nozzle but after growing into particles yield solid berfasa movement began dipengaruh gravity so that there is a downward movement. If the Crystal yield can be described in the form of a cylindrical sheath segment yield- SSY, SSY it will form a number of moving towards a joint forward-forward-downward and downward. With the movement of the early SSY SSY will preclude subsequent inception. Prior formed next SSY, SSY initial bottom collide with particles of an alkaline solution while the upper SSY particles collide with particles of precursor solution. Given SSY particle mass larger than the mass of the precursor solution and the reactants then eventually SSY will be able to exert a precursor solution and particle growth next phase is in the position of precursors and reactant particles erratic .. The formation of further particles within laminar flow conditions no nucleation process is not as fast and nucleation process SSY stage. The formation of the second phase nucleation and so on will be random in other words do not form a perfect SSY second stage.

For these reasons, it is recommended that the diameter of the nozzle is not too large, but also not too small (optimal diameter) in order to be more homogeneous particle formation.Nozzle shape radial models needed to accelerate the process of nucleation. In this model the formation of wrap nucleus formed in the direction perpendicular to the axis. The model can be created by placing a few pieces of the wall of the injector nozzle in a radial position of the axis of the injector.

As the initialization process X0.5kei measurement determined by reference parameter  $\rho$ , QE,, Mw, and R, by the following equation. X0.5.i value at constant PHF = 0.5 will depend on Ti and zi. Setting Zi based on the position of X0.5, i and has a pH value = pH0.5 as the original (value pH0.5) constant. Given the value of T allowing changes while the value of the position X0.5, I have determined there will be a change in the value of Zi is quite significant.

Fraction Yield based pH of the solution before and after the formation of crystals is expressed by the equation;

$$f(t) = \frac{10^{-pH_0} - 10^{-pH_t}}{10^{-pH_0} - 10^{-pH_t}}, f(t) = \frac{10^{-pH_0}}{10^{-pH_0} - 10^{-pH_0}} - \frac{10^{-pH_0}}{10^{-pH_0} - 10^{-pH_t}}.10^{-pH_t}$$

$$f(t) = M - N.10^{-pH_T}; \text{ with } M = \frac{10^{-pH_0}}{10^{-pH_0} - 10^{-pH_\infty}} \quad and N = \frac{1}{10^{-pH_0} - 10^{-pH_\infty}}$$
then

$$\frac{M - f(t)}{N} = 10^{-pH_T} \quad ; pH_t = \log\left[\frac{N}{M - f(t)}\right] \quad if \ f(t) = 0.5 \quad then \ pH_{t(0.5)} = \log\left[\frac{N}{M - 0.5}\right]$$
[1-4]

M and N depend on pH and temperature. Or pH liquid both depend on M,N and t finction.

$pH_0 \\$	pHt	М	Ν	pHx=1	pHt=0,5
12,6	11,87	-0,264110017	-0,083294218	-1,18117	-0,96254104
12,6	11,85	-0,249260175	-8,84408E+11	11,85	12,0720202
12,6	11,8	-0,216290421	-7,67427E+11	11,8	12,0299481
12,5	11,9	-0,335449831	-1,06079E+12	11,9	12,1037072
12,5	11,8	-0,249260175	-7,8823E+11	11,8	12,0220202
12,3	7,6	-1,9953E-05	-39811511,4	7,6	7,90102133
13	11,8	-0,067344911	-6,73449E+11	11,8	12,0744576

### **Stages Measurement;**

- a) As an initialization process X0.5kei measurement determined based on the reference parameter ρ, QE,, Mw, and R.
- b) The value of PHF = 0.5 x0.5.i the constant will depend on Ti and zi ..
- c) Setting Zi based on the position of X0.5, i and has a pH value = pH0.5 as before (value pH0.5) constant. Given the value of T allows changed while the value of the position of X0.5, I have

determined there will be a change in the value of Zi are quite significant.

Fraction Yield based pH of the solution before and after the formation of crystals expressed by Avrami equation as follows;

expressed by Avrami equation [3-4]

Examples of z and setting the table setting pH, innuendo obtained the activation energy of the linear graph X0.5 to 0.5 / z, as follows.

pH <sub>0</sub>	pHt	М	Ν	pHx=1	Setting pHt=05	Z [CC/dt]	X0.5	L=Ln X0.5
12,55	11,87	-0,264	-0,08329	-1,181	-0,962541			
12,55	11,85	-0,249	-8,8E+11	11,85	12,07202			
12,55	11,8	-0,216	-7,7E+11	11,8	12,029948			
12,55	11,9	-0,288	-1E+12	11,9	12,113294			
12,55	11,8	-0,216	-7,7E+11	11,8	12,029948			
12,55	7,6	-1E-05	-4E+07	7,6	7,9010251			

Note.

Setting is done by regulating the discharge pH z, the pH is read from the pH meter with the one shown in the above table. Create a graph to  $Ln \times 0.5 (\times 0.5 / z)$ . The coefficient value will be tangent direction of the chart and the activation energy value will be crystal formation ..

# I. Configuration Synthesis Reactor Flow Injection.

The third reactor limitation above can be filled with liquid reactants reacting in a narrow space and flow, so that the onset of the nucleation process is not to be the same but the age of the nucleus particles and the growth environment can be conditioned -growth same nucleus.

The synthesis process is a method of synthesis of Flow Injection Synthesis- FIS, the liquid reactant material is injected into the flow of the relative speed laminar alkaline solution. T laminar flow may occur in pipes or hoses with a relatively small diameter orifice or a pipe that has undergoing the process of deflection. For this purpose can be made form the flow in the hose is wound in a coiled shape. End of the first coil as the position of the injector while the other end of the coil as a restraint on the growth of particles. In the form of a liquid surfactant administration dispersants or alternating magnetic behavior. In the diagram FIS reactor configuration is described as follows;

# Figure reactor with FIS technique

Methods FIS will generate nucleation process sequence as a continuous, although the nucleation process itself occurs descret per particle, but it can condition the age of nucleation and growth of particles of uniform by means conditioning the fluid flow velocity solution reactant and the alkaline solution is constant, and minimize the growth process the particles on Curing Bath has;

• pH = 8.5 lower than normal reaction pH around 13.

• Temperature 250C lower than the reaction temperature of about 350C.



## Figure Coil of FIS in half time Process.

In order to obtain estimates of the time and speed of the process required a general description of synthesis process kinetics characteristics of conventional coprecipitation, such as activation energy, half-life of the coprecipitation process, etc.

Figure stages motion pulse suppressor cylinder head flexible hose diameter d



Figure 3. Sketchy part of the rotor cylinder and cylinder head peristaltic pump pulse suppressor.

II.1 Energy Levels Precursor Solution and Solution precipitants.

The reaction between the solution of precursors with the precipitant will absorb or release energy in their environment that is the solution. Will change the energy level of the solution. If the nature of the exothermic reaction the temperature of the solution will increase, whereas if the reaction temperature endotermal then the solution will decrease.

According to a Van't Hoff, the effect of temperature on the solubility is expressed as follows:

$$\frac{dLn S}{dT} = \frac{\Delta H}{RT^2}, \quad \int_{\ln S1}^{\ln S^2} dLn S = \int_{T1}^{T^2} \frac{\Delta H}{RT^2} dT$$
[[InS2-LnS1]] =  $\frac{\Delta H}{R} (T_1^{-1} - T_2^{-1}) = Ln \frac{S2}{S1}; \quad Ln S = \frac{-(\Delta H)}{RT} + Tetapan$ 

In terms of :

1. S1, S2 = solubility of each - each substance at temperatures T1 and T2 is expressed in units of [g / 1000gram solvent], So for pure solvent = water, 1000 grams = 1 liter, 1 gram = mol x M.

 $M = \Sigma g / mol$ , so that  $S = M \ge 10$ -pOH

2. The dissolution  $\Delta H$  = heat (hot dissolution / g (g)).

3. R = general gas constant.

Obtained  $S1 = M1 \times 10^{-pOH1}$  Ln  $S1 = Ln M1 + Ln 10^{-pOH1}$ 

Ln S2= Ln M2 + Ln 10<sup>-pOH2</sup>

 $LnS2-LnS1 = Ln M2-LnM1 + Ln10^{-pOH2} - Ln 10^{-pOH} = Ln M2/M1 + Ln 10^{(pOH1-pOH2)}$ 

or  

$$Ln\frac{M2}{M1} + Ln10^{(pOH1-pOH2)} = \frac{-(\Delta H)}{R}(T_1^{-1} - T_2^{-1})$$

For this type of dissolved materials do not change, then the M2 / M1 = 1 Ln M2 / M1 = 0. Was obtained

$$Ln10^{(pH2-pH1)} = \frac{-(\Delta H)}{R} (T_1^{-1} - T_2^{-1})$$

To PH2 = pH ahir = pH $\infty$ , PH1 = initial pH = pH0 Then

$$Ln\{10^{(pH_{\infty}-pH_{0})}\} = \frac{-Q_{solution}}{R}(T_{0}^{-1}-T_{\infty}^{-1}) \qquad \dots 3$$

Q solution as changes in the level of energy in the solution.

In general the dissolution energy is positive (endothermic), so that by Van't Hoff, the higher the temperature, the more soluble substance. As for the substance - a substance that negative dissolution heat (exothermic), the higher the temperature the more reduced the soluble substance

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II.2 Mechanism Mode reactant stream streaming solution Precipitant and Precursors.

Laminar and turbulent flow mode adjusted to the yield of particles in solution. Reactant and precursor solution in the container vessel has a saturated or supersaturated solution. When the two solutions into inector batch had an ocean ready bereakksi.

To obtain a reaction optimal- reaction that enables yielding flawless, then both the reactant solution and the precursor solution requires turbulence mode in order to form the nucleus of the nucleus yields.

II.3 Planning Long Flow In Section Half Time Yield.

Physically crystal growth to achieve a fraction yield 50% disbursed in regularly hose wound on cylindrical tubes with diameter of about 20 cm. The length of the hose is designed in accordance with the flow rate (discharge solution yield) and a 50% fraction of the formation of crystals yield (half-life t0.5). For determine the half-life requires parameter data precipitant solution pH, pH of the solution yields at any time up to the steady state.

Parameter pH when the yield reaches 50% or half of the overall yield of the parameter data required initial pH, pH steady state, as well as a number of IPM parameter data in the time interval between the beginning until a steady state pH.

In this case illustrates the pH pHawal precipitant before reacting with the precursor, pH describes the pH precipitant steady state after reacted with precursors.

Yield graph formed against the formation time t is obtained as a function of concentration precipitant concentration changes as follows;

$$\% Yield = \frac{\Delta Cpr_{setiap \ saat}}{\Delta Cpr_{sempurna}} = \frac{Cpr_{awal} - Cpr_{t}}{Cpr_{awal} - Cpr_{sst}} \qquad \dots 4$$

considering that pH = -log (H + concentration), pOH = -log (concentration OH-)then for the precipitant solution is an alkaline solution (NaOH), where Na (OH) = Na + + OH-, concentration OH- = concentration of NaOH, obtained that -log (OH- concentration) = - log (concentration of NaOH) = pOH.

So that the concentration of NaOH = 10-pOH,

Cp previous = 10 –pOH prvious, Cprt = 10-pOHt, Cprsst = 10-pOHsst [7,8] The fraction yield

$$\% Yield = \frac{Cpr_{awal} - Cpr_{t}}{Cpr_{awal} - Cpr_{sst}} = \frac{10^{-pOH_{awal}} - 10^{-pOH_{t}}}{10^{-pOH_{awal}} - 10^{-pOH_{sst}}} \dots 5$$

pH at fraction 50% yield can be obtained by the equation

To obtain a fraction of the time requirements yield of 50%, required observation time table, pHawal, pHsteady state, and the fraction of the yield. It can be said that the initial reaction is necessary to perform the synthesis of small amounts of reactants injector and small elemevyer glasses. From the list

will be obtained fitting fraction half-life t0.5.

Eg long range X0.5 flow [m] with a peristaltic pump discharge capability netto z [m3 / sec], the flow cross-sectional area A [m2] then the range is x0.5, X0.5 = (z. T0.5) / A

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At the position of X0.5, placed electrode pH meter.

In the same way the yield fraction X0.5 list can be used to determine the fraction of the time required to reach 100% yield, which is characterized by a pH of data that shows the state mantab (unchanged) or steady state pH. So Xsst = (z.tsst) / A.

As technically the most convenient position rejoins. hose by wrapping in a cylindrical tube with a diameter that is not too small to avoid the accumulation of hose, which will cause the precipitation process yields occur prior to the end to yield .

Optimization election sectional area of the inlet hose formulated apart from the provisions of the flow rate, the length of the channel, and time but also need to pay attention to the activation energy coprecipitation reaction process. Optimization parameters of these parameters can be obtained from the

initial experiments (initialization preparation).



series of 2 coil hose within half.

11 of the equation, there is a relationship between t0.5 parameter with the particle growth rate;

With in  $\Phi$  d particle diameter , N=6.25 x 10<sup>23</sup>

$$\phi_p = 0.001864 \left[ \frac{z.t}{A.X_{0.5}} \right]^{1/3}$$
.....8.

Was obtained provisions of the parameters that determine the particle size of the ocean inlet hose dimensions yield among others; sectional area of the channel, the channel spacing when the half-life and flow. the greater the hose cross-sectional area smaller the particle radius (particle diameter) or particle size yields.

$$A = 0.001864 \left[ \frac{z.t}{\phi_p.^3 X_{0.5}} \right] \qquad \dots 9.$$

t max = 2 x t0.5

 $A_{\max imum} = 0.001864 \left[ \frac{z.2t_{0.5}}{\phi_p.^3 X_{0.5}} \right] \dots 10$ 

If the surface area of the initial flow parameters as a standard in the estimation of particle size then apply the equation;

From the equation above shows that the minimum size of particles occurs if the maximum flow surface area expanded.

Example of a cross-section Hose From pH Data Logger Debit and data solution Specific precipitants On X0.5 position.

	pHset	Z		
No	0.5	[m3/det]	X0.5	Ln(x0.5/z)
1	11.85	1.5E-06	3.5	14.6628
2	11.85	1.40E-06	3	14.5777
3	11.85	1.60E-06	3.8	14.6805
4	11.85	1.80E-06	4	14.614
5	11.85	1.90E-06	4.1	14.5846
6	11.85	2.00E-06	4.05	14.5211

From the equation of the equation, Ln X0.5 / z = (1 / k) X0.5 - A Ln

And data Ln X0.5 / z, and X0.5 create linear graph. Sarikan graph linear equations are concerned. Liken the theoretical equations are obtained slope and insept. as follows;

 $\ln X0.5 / z = -0.0223 X0.5 + 14.69$ 

1 / k = - 0.0223 ----> k = -44.84

 $\ln (A) = -14.69 - A = 0.4 10^{-7}, -7 = 0.367 \text{ m}$  $pH_0$ pHt Μ Ν pHx=1  $pHt_{=0,5}$ 12,6 11.87 -0,264110017 -1,18117 -0,96254104 -0.083294218 12,6 11,85 -0,249260175 -8,84408E+11 11,85 12,0720202 12,6 11,8 -0,216290421 -7,67427E+11 11,8 12,0299481 12,5 -1,06079E+12 11,9 -0,335449831 12,1037072 11,9 11,8 12,5 12,0220202 -0,249260175 -7,8823E+11 11,8 12,3 7,6 -1,9953E-05 -39811511,4 7,6 7,90102133 13 11.8 12,0744576 -0,067344911 -6,73449E+11 11.8

## II. Conclusion.

- Flow Injection Synthesis has the same purpose, ie reacting and conveying the reaction result through the closed channel in the form of a capillary pipe. In the model presented more emphasis on the process has been reacted. The reaction is carried out on the injector, where the acid and base are reconciled.
- The length of the Flow Injection Synthesis trip is estimated to be equal to the reaction time of the reactant. The reaction product is collected in a container, cleaned and diluted so that the pH of the solution is = 7, Then it is precipitated and dried.
- The separation of the yield to be filtered and deposited approximately 4 to 5 times, using another container.

# Reference.

- [1]. Avrami, M (1939),. Kinetic of Phase Change I. General Theory., Journal of Chemical Physics 7 (12): 1103-1112.2
- [2] . Avrami, M (1940),. Kinetic of Phase Change II. Transformation-Time Relations for Relations for Distribution of Nuclei., Journal of Chemical Physics, 8 (2): 212-224.
- [3]. Avrami, M (1941)., Kinetic of Phase Change III. Granulation, Phase Change, and Microstructure., Journal of Chemical Physics 9 (2): 177-184
- [4]. J. Vazquez, P.L. Lopez-Alemany, P. Villares, R. Jimenez-Garay., Generalization of the Avrami equqtion for the Analysis of non-isothermal Transformation Kinetics. Application to the Crystallization of the Cu<sub>0.20</sub>AS<sub>0.30</sub>Se<sub>0.50</sub> alloy., Departamento de Fisica de la Matera Condensada, Facultad de Ciencias, Facultaded de Cadiz, Apartado 40, 11510, Puerto Riel, Cadiz, Spain
- [5]. H.A. Hashem<sup>a,1</sup> and S. Aboulhassan<sup>b</sup>., Non-Isothermal Cystallization Kintics Of Sb<sub>10</sub>Ge<sub>10</sub>Se<sub>80</sub>
   Chalcogenide Glass., <sup>a</sup>Physics Department, Faculty of Science, Zagazig University 44519, Egypt. <sup>b</sup>Physics Department, Faculty of Sciences, Benha, Egypt.<sup>1</sup>E-mail address: <u>ha hashem@zu.edu.eg</u> FIZIKA A (Zagrep) 15 (2006) 4, 237-250.
- [6]. R.Ahmadi, H.R. Madaah Hosseini and Masoudi., Avrami Behavior of Magnetite Nanoparticles Formation in co-precipitation Process., Department of Materials Science and Engineering, Sharif University of Technology, P.O.Box 1458889694, Tehran, Iran.
- [7]. Boryana D. Koleva<sup>a</sup>, and Elisavta H. Ivanova<sup>b</sup>., Flow Injection Analysis with Atomic Spectrometric Tetction.(Review Article)., <sup>a</sup>Directorate General, National Centre of Metrology, Bulgarian Institute of Metrology, G.M. Dimitrov Blvd 52 B, Sofia, Bulgaria. <sup>b</sup>Institute of General and Inorganic Chemistry, Bulgarian Academic of Sciences, Acad. G. Bontchev Str.Bl 11,1113 Sofia, Bulgaria
- [8]. <sup>a</sup>Mingjun Yuan, <sup>a</sup>Lih-sheng Tung, <sup>b</sup>Daniel F. Caulfield ., Cystallization and Thermal Behavior of Microcellular Injection-Molded Polyamide-6 Nanocomposites., <sup>a</sup>Polymer Engineering Centre, University of Wisconsin, Madison, Wisconsin 53706, <sup>b</sup>USDA Forest Products Laboratory, Madison, Winconsin 53726.
- [9]. Takeo Ozawa., Thermal Analiysis- Review and Prospect., Thermochimica Acta 355 (2000) 35-42. Chiba Institute of Technology, Tsudanuma 275-8588, Japan, <u>www.elsevier.com/locate/tca</u>.
- [10]. Erh-Chiang Chen, Tzong-Ming Wu., Nonisothermal Crystallization Kinetic and Crystalline Structure Of Nilon 6/ Funtionalized Multi-Walled Carbon Nanotube Composite., Depatment of Material Science and Engineering, National Cung Hsing Unversity, Taichung, Taiwan.

- [11]. Chen Shouxi., A Study On The Crystallization Kinetic Of Nylon, Insttute of Chemistry, Academia Sinica, Beijing, Chinese Journal Of Polymer Science, Vol 15, No 4, 1997.
- [12]. Zhang Zhiying ., Theoretical Analysis of Kinetics of Nonisothermal Cystallization of Polymer., Department of Textile Chemical Engineering, Tianjin Institute of Taxtile Science and Technology, Tianjin 300160).
- [13] Yi Wu, Xian-yuan Liang, Rui-fen Chen, Yong-gang Shangguan, Qiang Zhen., THERMAL AND RHEOLOGICAL BEHAVIOR., *Chinese Journal of Polymer Science*. 2012 30 (3): 470-477. (Papers) DOI: 10.1007/s10118-012-1145-z.