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Indirect Validity Test of Measurement Instrumentation in Flow Injection Synthesis Technique of Cu-Zn Ferrite Formation Process

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Abstract

The Flow Injection Synthesis-FIS instrumentation measurement system has to validity test either will be used by workers or for the instrument validity test. Repeatability test is one of easy method to do indirect validity test. The test is done besides of the instrumentation data test but also product consistency test and production system. All of the measurements systems that will be tested have to fulfill requirement as valid and reliable measurement instrumentation. In this work six sample of X-Ray Diffraction test divided in two groups of temperature different have been prepared for repeatability test base in the yield of FIS process of Cu Zn Ferrite synthesis. All of the processes results have to validity test to convince the quantitative product analysis. The indirect test at six yields sample show high repeatability with atomic content deviations less than 2% but in relative validity, fulfill requirement product reproducibility workers. The FIS Instrumentation system include FIS reactor, pH data logger, peristaltic pump which its use in the non-isothermal reaction.

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Keywords: Flow Injection Synthesis (FIS); Non-Isothermal; Avrami methods; FIS Instrumentation; Reproducibility; Valid; Reliable; Data logger.

1. Introduction

This paper is one of the essence of dissertation writers to obtain a doctorate in the field of material titled Synthesis and Determination of Energy Forming CuZn Ferrite materials using techniques Flow Injection Synthesis-FIS [1].The FIS synthesis reaction is carried out in the adiabatic reaction batch where in this method the reaction analysis carried out on non-isothermal systems and usr Avrami-Ozawa-Kissinger equation [2]. The special characteristic of the non-isothermal FIS were both temperature and pH parameters changed regularly to keep pace with the flow of the reactants. In the especially case the yield ferrite in this research is compulsory to proof that yield of FIS process is uniform although in different time of process where the several ferrites are developed with both the estimation of yield uniformity and reproducibility control based on manipulation of the processing parameters [3]. The yield in this work is specific Ferrite $Cu_{(1-x)}Zn_x$ Fe_2O_4 use salt of chloride acid and alkaline base raw materials [4,5]. The question arises whether the reaction proceeds uniform or not, especially the chemical composition of Cu-Zn Ferrite. One of the simple proof are both repeatability and reproducibility test such as answer in this work. In general, wet chemical deposition synthesis methods together - co-precipitation forming materials such as iron oxide; -Magnetite Fe3O4, MeFe2O4-ferrite with Me as a metal atom such as Fe 2+, Mn 2+, MI2 +, Zn 2+, Cu 2+ has many advantages, among others; is performed at room temperature, it is easy to generate yield with a high success rate, high yield homogeneity, including as nanomagnetik material that has properties of high reliability to produce a high degree of homogeneity yield [3]

The Magnetite and Ferrite as iron oxide materials are most popular as nano magnetic material easy to process in low temperature [4,5], either use mechanical process or wet chemical process.[5,6] where the raw materials will be reacted as co-precipitation in the bath reactor that will be measured all of reaction parameter by pH Data logger automatically.

One of the co-precipitation method completed by measuring equipment of the process parameter devices is Flow Injection Synthesis-FIS technique such as have be done by worker Alvares [6] and Sasito [7] in both different method and different research worker for the reason of the yield materials should be used as product of process system, then there were compulsory to validity and reliability test of both process and measurement instrumentation use indirect test mainly either repeatability or reproducibility [1].

The FIS-apparatus is adapted to support the process in the non-isothermal should be able to adiabatic maintain. The Piping system of fluid flow and using of the peristaltic pump following a pump electronic control circuit for use is an absolute terms. Using of data logger which use Sim Drive card will provide easier data processing..

The Cu-Zn Ferrite material is one of mixing ferrite represent an important class of the functional magnetic materials, largely used in electronic industry, catalytic, CO gas oxidant, inductor of Electromagnetic Interference Suppression-EMI and many other fields of interest [6] can be FIS synthesized .

The main purpose of the validity testing in all aspect of using either in the previous measuring instrumentation or in the material production process, were to prove whether the material produced in the same technique synthesis will give the uniform product. Then convinced the results of the processed parameter measurements are valid and reliable. In the in-direct test, the worker just for computing reproducibility or repeatability in standard deviation which it is result of derivation. In the one of the Flow Injection Synthesis (FIS) the parameter process can be used for controlling of the rate reaction process, especially in every process rate almost no excess to homogeneity of yield then the FIS process will be able as reproducible reactor.

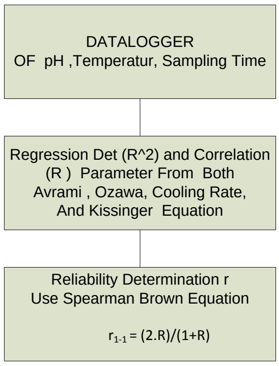

Figure1: The diagram of calculation step to determine both the direct validity and reliability test.

This evident will be proved that using variant of analysis method as results measuring of a lot of measurement device such as thermometer, pH meter, timer, X-Ray diffraction and other equipments considered to have been both valid and reliable [1] caused all of measurement device truly have been validity tested by manufacturing company, but in one measuring unity test validity has to indirect measurement mainly product uniformly test to see production aspect such as worker, environment, method of production and etc. If all aspect in the same condition but existing additions treatment such as annealing allows the change of the chemical structure of materials so that the measurement results can be invalid and should be avoided.

1.1 Cu-Zn Ferrite Production Use Flow Injection Synthesis Background

In the Flow Injection Synthesis-FIS method the reactants were injected to the solution flow in the reactor basin (Flow Injection Synthesis in Bath) Will obtains the yield has appropriate of the parameter process. There are two types FIS, mainly Isothermal and non-isothermal. The Isothermal type of FIS used to produce of yield besides uniform of the chemical composition but also uniform particle size mono-disperse. The Non-isothermal FIS type besides used to produce of uniform chemical of yield but used to determine crystallization energy of yield. The relationship between yield fractions with pH of the reaction expressed by the equation

$$Y(\%) = \frac{(10^{-pH0} - 10^{-pH_t})}{(10^{-pH0} - 10^{-pH_\infty}}.$$

where pH_0 as previous pH, pH_t as real time pH, pH_∞ as steady state pH of yield solution [1,3,6].

Using of the flow injection process usually use the measuring instrumentation and process tool to determine of process parameter such temperature, pH and the long time of process.

The Conditions for specific repeatability of the sample test precision of weather laboratory , operator, laboratory, apparatus are the same but time between test need several days not short time such as normally repeatability. Then combine of two repeatability test in different working time be a reproducibility test then may as relative validity test [7].

One of the validity test method is repeatability test. The advantage of the method are simple, minimum amount of sample able to take six data in different time but in one series of process, able to use to determine the homogeneity of the sample matter, able to validity test both instrumentation measure and product quality.

In the present work, need both X-ray diffraction characterization and Rieldvelt refinement analysis to exposure the atomic composition of $Cu_{(1-x)}Zn_xFe_2O_4$ as yield of reaction between raw material $FeCl_2.4H_2O$, $FeCl_3.6H_2O$, $ZnCl_2$, $CuCl_2.2\ H_2O$ and Na(OH) solution. [10], which attention to the Ferrite material yield include has a thermoelectric properties and chemical properties of the catalyst as a result of the Jahn-Teller effect[10*]. The effect should be the attractiveness on the synthesis of co-precipitation at low cost [11,12].

1.2 The equipment and measuring Device

In this study, there was some process equipment functions simultaneously as the measuring equipments have been validity test by the product company such as; pH, temperature, pH Data logger.

The list of the Non-Isothermal FIS equipment [1] as below;

1) Adiabatic Bath reactor.
2) Solution of alkaline tank .
3) Hose and injector system.
4) Peristaltic pump Electric stirrer.
5) pH and Temperature data logger.
6) SD Card.
7) pH sensor an PTC temperature sensor.
8) Variable DC power supply.
9) Pump speed controller.

All of the FIS equipment arranged as the following figure2.

2. Experimental Details

In this study, using X-ray diffraction is absolutely necessary to elaborate CuZnFerrite characterization. Mainly used to determine chemical composition of products synthesis by FIS method. To obtain the results of a careful calculation of chemical composition used Rieldvelt refinement methods [1,11].

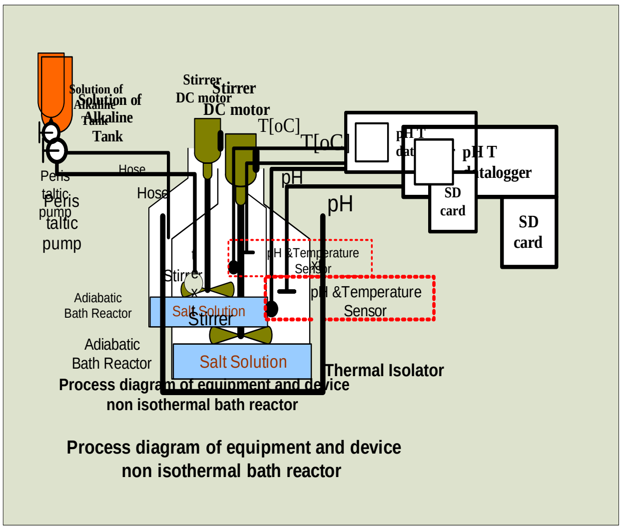

Figure 2: Schematic illustration of the Non-Isothermal FIS bath reactor of co-precipitation using thermal insulator as jacket of bath [1].

2.1 The Raw Material Synthesis.

The $Cu_{(1-x)}Zn_xFe_2O_4$ (**0.35<x<0.45**) was synthesized by co-precipitation method using Flow Injection Synthesis technique at the same parameter process but in the different sampling time. The Alkaline solution Na(OH) were flowed by peristaltic pump and were injected to the mixer solution of both $FeCl_2.4H_2O$, $FeCl_3.6H_2O$, $ZnCl_{2,}$ and $CuCl_{2.}2\ H_2O$, **[10]** in the adiabatic batch reactor.

The series of sampling time were figured in table 1;

Table 1: The Series of the sample code and notification.

Sample Code	Sampling time	Notification	Date
CZF A.3	1. minute	Project A	Mei 2014
CZF A.6	2 . minute	Project A	Mei 2014
CZF A.9	3. minute	Project A	Mei 2014
CZF B.4	1.2 minute	Project B	June 2014
CZF B.7	2.3 minute	Project B	June 2014
CZF B.10	3.8 minute	Project B	June 2014

All of the sample after finishing precipitation reaction in alkaline condition as fast as allow cleaning-precipitating process, use and dissolve in ion-free water one by one step until pH of solution of yield close to seven . The next step is to dry of yield both project A at 70 ^{0}C and project B at 200 ^{0}C. See figure 1 that crystal peak Cu-Zn Ferrite will start at 97 ^{0}C without influence to the atomic distribution.

2.2 Characterization of The Samples of Cu-ZnFerrite

The Synthesized samples were characterized by powder X-ray diffraction using a Siemens diffractometer machine. The XRD data were recorded use Cu K_α radiation where λ=1.5406 Å. The range of intensity distance 2θ of $10^0 - 80^0$ goneometer . The atomic material content no exchange in the heat treatment below melting point this reason be a reference on the uniformity of this research will take place at the co-precipitation reaction.

Heat treatment will result the higher material density, such as in the figure 2 below where material before and after heating look very different in the X-Ray peak. .The patterns of X-Ray Diffraction first group of test samples (ZCF1.3, ZCF1.6, ZCF1.9) as shown in Figure 3 pattern top, indicates that the material is not structured (amorphous). While the diffraction pattern of the second group of test samples (ZCFB.4, ZCFB.7, ZCFB.10) is presented in Figure 4.2, 3 bottom pattern visible presence of several diffraction peaks spread of the scattering angle 2θ> 20 ° to 2θ <70 °

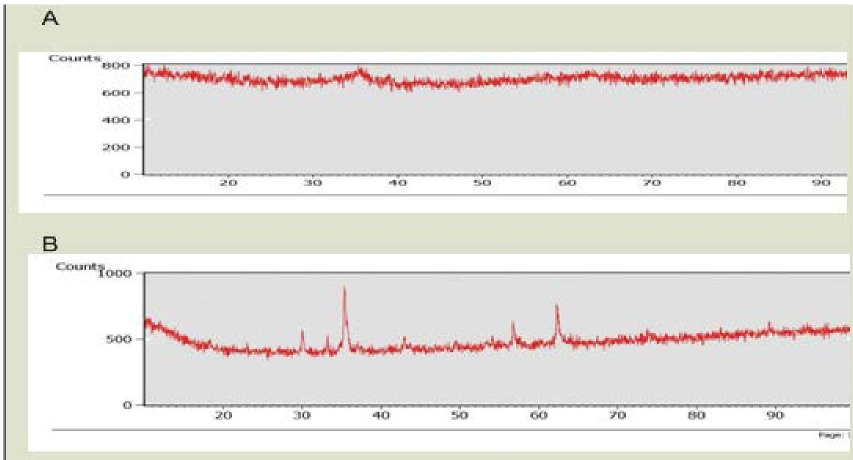

Figure 2. a.The XRD pattern of CuZnFerrite yield powder have heated to 200 ^{0}C, b. heated to 700 ^{0}C

In the figure 2, have seen the different of between the XRD pattern of CuZnFerrite yield powder have heated to 200 ^{0}C , and . heated reach to 700 ^{0}C . The peak of second pattern where have to heat treatment specified to see truly pattern of the Cu-Zn Ferrite, but not to explore the physical characteristic, cause any exchange to dimension and particle density.

The X-Ray Diffraction pattern tend as non-crystalline powder.

Diffraction pattern of the first group of three samples (ZCFA.3, ZCFA.6, ZCFA.9) and three samples of the second group (ZCFB.4, ZCFB.7, ZCFB.10) have a common approach CuZnFerrite diffraction pattern. The second group through the process of heat treatment to bring a more clear diffraction peak. the conditions of each process establish of repeatability such as the similarity of experimental tool observer, measuring instrument, laboratory conditions objective ,repetition over a short period of time but process A to process B do not continue in repetition over a short period of time or the sampling time does not exceed one day hen the process as in the Intermediate Precision Condition [6].

Base on the similarity diffraction patterns and the use of methods of smoothing-Rieldvelt refinement. in the analysis of the peak information can be obtained. The results of X-Ray Diffraction identification were obtained as profiles follows in the test sample; (ZCFA.3, ZCFA.6, ZCFA.9,ZCFB.4,ZCFB.7,ZCFB.10) as figure 1, were presented of samples for validation testing as mentioned above. The first group of test samples placed bottom, group both at the top. The sampling time of 21 days, CZFA.3→CZFA.6→CZFA.9 need 2 sec each part, CZFB.4→CZFB.7→CZF1.-0 need 2 sec each part. The X-ray diffraction profile of the top to down with ZCFB.10 code, ZCFB.7, ZCFB.4, (who are members of the group 2) and ZCFA.9, ZCFA.6, and ZCFA.3.

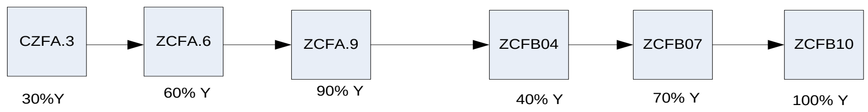

Figure 2: Map sampling from process A to process B

which is where the first group was given no heating while the second group was given heating at 200 oC for 2 hours

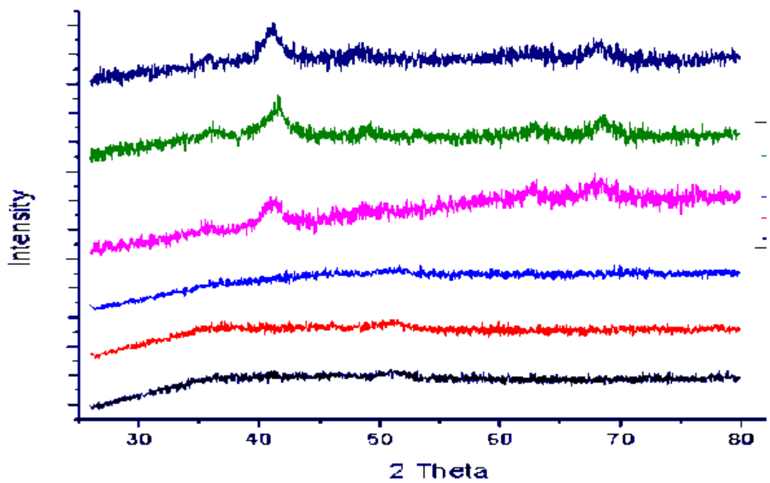

Figure 3: The x-ray diffraction pattern of sample Zn- CuFerrite Six variants of the test sample.

The FIS process which is most easily and effectively generate yield with relatively small particle size of the nano-meter sized. LaMer and 1950 Dinegar create the diagrams that can be used for controlling synthesis co-precipitation process making it possible to generate relatively complete of yield then the yield tends to poly-disperse [4,12] but there were still uniform in chemical composition .

The Cu-ZnFerrites or $Cu_{1-x}Zn_xFe_2O_4$ material were synthesized by wet-chemical co-precipitation. The result of XRD refinement may continuously done until GOF around 1 [11]. In this research result of refinement such in both table 2.1 and table 2.2 below;

Table 2.1: The Results of Rietveld Refinement Lattice parameters, Cationic Distribution the First Group Test Cu-Zn Ferrite Material

Sample Code	ZCFA.3	ZCFA.6	ZCFA.9
pH	*13.0*	*12.17*	*13.37*
R factor	*w*Rp = 4.08 Rp = 3.24	*w*Rp = 3.91 Rp = 3.07	*w*Rp = 3.90 Rp = 3.10
space group	F d -3 m (227)	F d -3 m (227)	F d -3 m (227)
Lattice Parameter	a=b=c=8.287(6) Å $\alpha = \beta = \gamma = 90^o$ V = 569.2(1) $Å^3$ ρ=5.398 $gr.cm^{-3}$	$a = b = c$ = 8.501(4) Å, $\alpha = \beta = \gamma = 90^o$ V = 614.4(1) $Å^3$ ρ = 5.166 $gr.cm^{-3}$	$a = b = c$ = 8.563(4) Å, $\alpha = \beta = \gamma = 90^o$ V = 528.0(1) $Å^3$ ρ = 5.234 $gr.cm^{-3}$
Cationic composition	Zn =0.4698 Cu =0.2522 Fe =2.2768	Zn=0.4719 Cu=0.2492 Fe =2.2842	Zn=0.47460 Cu=0.2546 Fe =2.2848
Empirical Formula	$Zn_{0.47}Cu_{0.25}Fe_{2.28}O_4$	$Zn_{0.47}Cu_{0.25}Fe_{2.28}O_4$	$Zn_{0.47}Cu_{0.25}Fe_{2.28}0_4$

Table 2.2: The Results of Rietveld Refinement Lattice parameters, Cationic Distribution the Second Group Test Cu-Zn Ferrite Material

Sample Code	ZCF-B.4	ZCF-B.7	ZCF-B.10
pH	*13.0*	*12.17*	*13.37*
R factor	*w*Rp = 3.96 Rp = 3.13 χ^2-chi-squared = 1.16	*w*Rp = 3.81 Rp = 3.00 χ^2-chi-squared = 1.06	*w*Rp = 4.08 Rp = 3.23 χ^2-chi-squared = 1.06
Space Group	F d -3 m (227) Cubic	F d -3 m (227) Cubic	F d -3 m (227) Cubic
Lattice parameter	$a = b = c$ = 8.40 Å, $\alpha = \beta = \gamma = 90^o$ V = 593.8(2) $Å^3$ □ ρ = 5.271 $gr.cm^{-3}$	$a = b = c$ = 8.39 Å $\alpha = \beta = \gamma = 90^o$ V = 591.9(3) $Å^3$ ρ = 5.264 $gr.cm^{-3}$	$a = b = c$ = 8.37 Å $\alpha = \beta = \gamma = 90^o$ V = 587.1(3) $Å^3$ ρ = 5.171 $gr.cm^{-3}$
Cationic Composition	Zn =0.4828 Cu =0.2623 Fe =2.2549	Zn = 0.4846 Cu = 0.2667 Fe = 2.2487	Zn=0.4882 Cu=0.2726 Fe =2.2392
Empirical Formula	$Zn_{0.48}Cu_{0.26}Fe_{2.25}O_4$	$Zn_{0.48}Cu_{0.26}Fe_{2.25}O_4$	$Zn_{0.49}Cu_{0.27}Fe_{2.24}0_4$).

Table 3: The empirical formula of each Cu-Zn Ferrite sample and atomic fraction in 6 samples in different time of process.

No	Sampel Varian	The Empirical Chemical formula	Contents of the atomic fraction		
			Zn	Cu	Fe
1	ZCFA.3	$Zn_{0.426}Cu_{0.267}Fe_{2.307}O_4$	0.426	0.267	2.307
2	ZCFA.6	$Zn_{0.471}Cu_{0.249}Fe_{2.284}O_4$	0,471	0,24	2,285
3	ZCFA.9	$Zn_{0.474}Cu_{0.254}Fe_{2.285}O_4$	0,474	0,254	2,285
4	ZCF B.4	$Zn_{0.483}Cu_{0.262}Fe_{2.255}O_4$	0,483	0,262	2,255
5	ZCFB.7	$Zn_{0.485}Cu_{0.267}Fe_{2.249}O_4$	0,485	0,267	2,249
6	ZCFB.10	$Zn_{0.488}Cu_{0.273}Fe_{2.239}O_4$	0,488	0,273	2,239

From table 3 and the table 2 he Content of Cu, Zn, Fe of the Cu-ZnFerriteSample Material Retrieved the chemical compound average (uniform) as $Zn_{0,471}Cu_{0,220}Fe_{2,270}O_4$ The identification of all six variants of the above test samples with the some contents of the metal atom can be compound in tabulated 4. as follow;

The study of Ferrite material beside as of huge number of studies but also not well resolved problem [10] this is caused by easy of the ferrite material well performed at low temperature. The catalytic effect by the number of atomic material and physical properties of performed process make the number of Ferrite material established at any kind of properties. By using pH and temperature memory recording the trapping of material performed able to trace and reconstructed as new material. Getting of Both the X-Ray Diffraction characteristic of material genuine and Rieltveld refinement analysis is needed where no physical treatment for developing new material.

Table 4: The percentage of The Standard Deviation of the formation Error 6 Variants.

	Content of metal			Error / Deviation		
	Zn	Cu	Fe	ΔZn	ΔCu	ΔFe
ZCFA.3	0.43	0.267	2.31	-0.045	0.005	0.037
ZCFA.6	0.471	0.249	2.285	0	-0.013	0.015
ZCFA.9	0.474	0.254	2.285	0.003	-0.008	0.015
ZCFB.4	0.483	0.262	2.255	0.012	0	-0.015
ZCFB.7	0.485	0.267	2.249	0.014	0.005	-0.021
ZCFB.10	0.488	0.273	2.239	0.017	0.011	-0.031
Average						
			Total of	0.001	0.000	0
			Error Square	1E-06	0	0
	Total of error			(ΔZn)^2	(ΔCu)^2	(ΔFe)^2

	ZCFA.3	0.002	3E-05	0.001
	ZCFA.6	0	2E-04	2E-04
	ZCFA.9	9E-06	6E-05	2E-04
	ZCFB.4	0.0001	0	2E-04
	ZCFB.7	0.0002	3E-05	4E-04
	ZCFB.10	0.0003	1E-04	1E-03
Total square = 6 x (sub total square)		0.0027	4E-04	0.003
$6(\Delta x)^2$		0.016	0.002	0.021
STD		0.02308	0.009	0.0263
	Error	0.00942	0.0037	0.0107
% error		2.00	1.4006	0.4721

% error atomic content = (2% + 1.40 % + 0.47 %) / 3 = 1.29 %

From Table 2 the Cu-Zn Ferrite atomic contain averages were obtained both the elements Zn = 0.471, Cu = 0.262, and Fe = 2.270.

The percentage of the standard deviation of both the elements Zn = 0.023, Cu = 0.009 and Fe = 0.0263, with an oxygen atom as a guideline, which the atoms are considered to be irregularities. Uncertainties percentage of atomic elements Δy = Standard Dev. / $\sqrt{n}$. then $\Delta Zn = 0.023 / \sqrt{6} = 0.00942$ (2%), $\Delta Cu = 0.0037$ (1.4%) ΔFe = 0.011 (0.47%). Average the percentage of deviation composed of all the atomic ZnCuFerrite close to 1.29%. Based on the deviation of the atomic material so obtained compound ZnCuFerrite average Zn=0,471 Cu=0,262 Fe=2,270 O=4 with a maximum deviation rate of around 2%, the deviation is still allowed in advance research where the error less then 5%. Thus the sixth variant of the test sample have valid and reliable research data.

3. Discussion

According to the results of the ferrite materials density of relating not only depend on the content of the material atoms but also depend on the environment material background, If it is warming up to 200 ^{O}C homogeneous results while for do not heating Ferrite material give a result in different density , the material must be in different back ground such of temperature, pressure, pH parameter. It is seen that the content of the atoms in a consistent uniform while the Ferrite density variations inconsistent in 3 variations. So that should be assumed that it is the nonlinearity of the material density of atoms containing may be caused by the temperature difference of the CuZnFerrite. The atomic content of yield the material will never change with heating the material to be changed is the physical characteristics of materials such as material density.

The result of the Rietveld Refinement of the X ray Diffraction give a good of fit-GOF or $\chi 2$-chi square close to one which illustrates that the data processing is in very good statistics, so can be expected to provide results of approached the value of the content of the atomic Cu Zn Ferrite materials are properly. This research proofs that Flow Injection Synthesis technique in bath reactor version as FIS Alvares version capable to result of homogeneous yield in high reproducibility [12]. The test of job on the project separately A and B respectively are repeatability, but from project A to project B is reproducibility. In this study both properties of repeatability and reproducibility which supported by adequate data [13] , then the project A and Project B tend to have high reproducibility .To obtain the atomic composition of ferrite materials one of methods used is the analysis of the Rietveld refinement of X-ray diffraction test, running continuously performed until a goodness factor close to one [14]. In this study, it absolutely must be done in order to obtain the composition of atoms with high precision.Using the “bottom-up” approach, as chemical methods can be used to synthesize uniform nano crystals with controlled particle size through both chemical nucleation and particle growth process [15] in this work only FIS method in Isothermal where the yield will have properties uniform both in chemical composition and physical characteristic. Profile XRD of the Cu-Zn ferrite resembles to the profile of Fe-Mn ferrite heated at a temperature of 90-95 0C for 30 minutes [16], this massage that Xray Diffraction may be resemble but it can be distinguished by Rietveld Refinement analysis.

4. Conclusion

1. To fulfill the principle of instrumentation that the use of valid and reliable the repeatability tests have been conducted product-yield 6 variant, the sample material CuZnFerrite elements that make up the material is homogeneous with the atomic content level deviation of about 1%. This action give suggestion that instrument as valid restriction instrument but still in high reliable.

2. The results of the heat treatment study to the power of CuZn Ferrite yield the does not affect to the atomics content of the CuZn Ferrite compound although X-Ray Diffraction graphs looks different enough. The Heating effect is useful to clarify the diffraction peak. This proves that the yield of the Flow Injection Synthesis is homogeneous or uniform.The Reliability and validity test in this work use indirect test by repeatability and reproducibility method make easy work of the worker to proof uniform of yield.

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