Flow Injection Synthesis of CuZnFerrite in the Circular Hose Tube Coil

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Abstract: **In the Nano magnetic material Usually the most emerging is the Fe2O3 magnetite compound, and Nano magnetics provide unique opportunities to address the fundamental limitations of traditional electronic materials diversifications, such as Ni, Co, Mn, Zn, has strength to magnetic force but not Cu mean they have combine ZnFe, or CuFe, mainly in partcle size. With proper preparation then the desired form of the compound can be realized. Just as the Flow Injection Synthesis model Circular Hose Tube Coil is capable of realizing CuZnFerrite.**

Keywords: **Magnetite, Nano magnetics, diversification, particle size, Flow Injection Synthesis.**

1. INTRODUCTION

Cu,Zn,Fe,O are the main raw material of Ferrite, where the main formula are Cu(1-x)ZnxFe2O4. The reaction of the Ferrite material is in the precipitation of the salt chloride and sodium hydroxide.

There are several research purposes, observe the process of forming CuZnFerrite material which is done by precipitation method. [1]

A lot of method have been developed to form of ferrite material such as; high- energy grinding, plasma flame pyrolysis, electro-chemical, high thermal evaporation, hydrothermal preparation, sole gel, co-precipitation and etc. The simplest and economical process is co-precipitation. One of advantage co-precipitation method is the process parameter can be observed easily[2].

2. EXPERIMENTAL DETAILS

The Injector parts diagram, conditioner laminar flow yield mainly as Flow Injection Ferrite Materials Synthesis, the section essential part consists of;

1) Inlet for saline.

2) The inlet for alkaline solutions.

3) The nozzle of 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 solutions as a precursor to the super-saturated solution of lye, which need to be considered, among others;

- Availability of the two reactants sufficiently.

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

- Substitution between segments that are continuous in serentah followed by nucleation and growth process completely, following the movement patterns as follows.
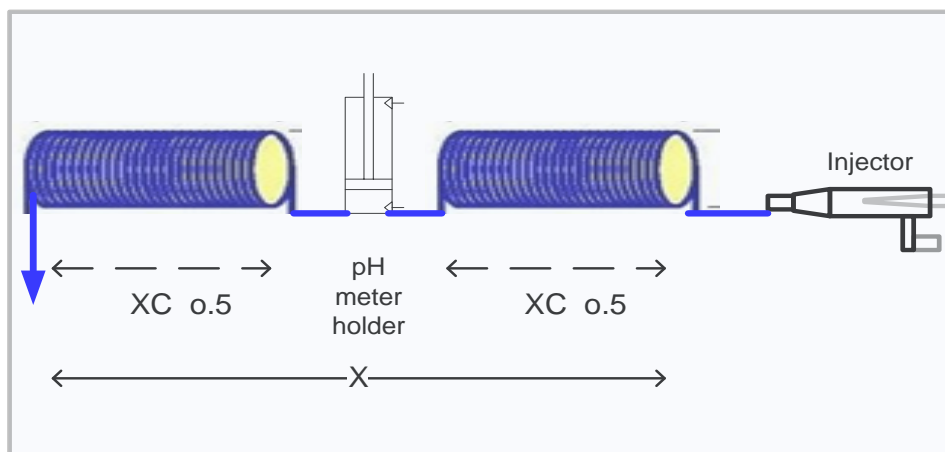

Figure 1. Injector and Circler Hose.

There are twou pipes inlet for reactans input mainly as salt solution and alkaline solution. pH salt is about 5 and alkaline is about 13. Mixturing of both In a 1: 2 ratio. Therefore with the ratio of the flow velocity equal, the speed ratio is also made equal.

Raw material,

- Precursor salt chloride; $FeCl_3.6H_2O$, $FeCl_2.4H_2O$, $CuCl_2.2H_2.O$, $ZnCl_2$. The ratio of the precursor material is adjusted to the table, depending on the mole ratio of each material. In this case eg in the case of salt precursor formation 38.7 ml based on the table is required $CuCl_2.2H_2O=2.044$ gr/20ml H_2O, $ZnCl_2=6.54$ gr/20ml H_2O, $FeCl_2.4H_2O$ gr/20ml H_2O, $FeCl_3.6H_2O$ gr/20ml H_2O, Total liquid precursor 80 ml is put into 250 ml bottle that have been equipped with a diverter set to the injector as shown below;

-Precipitated sodium hydroxide; NaOH.

Alkaline liquids are prepared by dissolving the NaOH salt into demineralized aqua (DM) water. Air Akua DM is heated 80 oC, and dissolve pure NaOH into it, In 1 dose of 800 ml +13.86 ml.(813,86 ml) ml is put into 1000 ml bottle that have been equipped with a diverter set to the injector as shown below;
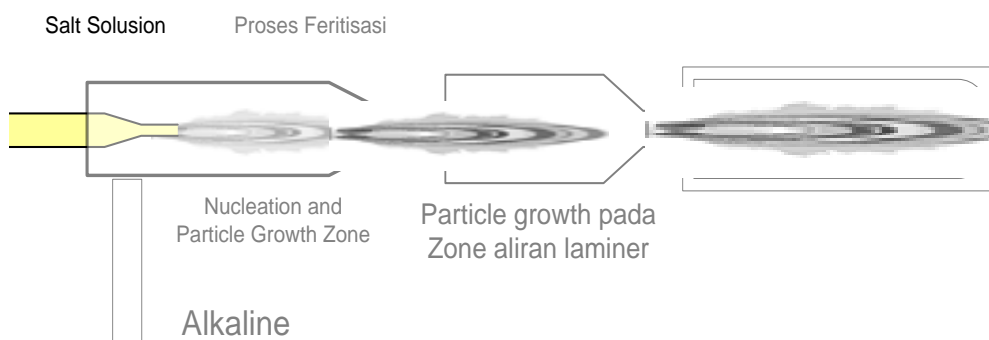

Figure2. Injector of Alkaline with salt precursor..

- Water Aqua De Mineralization (Aqua DM)

a. Main Equipment and Instrument as follows; 1. pH meters, 2. Thermometer,3. Demultiplex, Switching, 4. Data n Logger, 5. Injector and reactor, 6. Peristaltic pumps for acids and bases. 7. Batery 12 Volt, 8. Camera video, and 9.

Bucket Oven konvensional n the experiment, pH records with pH datalogger at two positions were performed using electronic position transfer (Demux Switching).

b. Expected product. The third solution of chloride salt in 60 ml of aquades is accompanied by a stream of nitrogen gas, to prevent the initial oxidation.   NaOH = 17grm / 600 ml aqua DM, pH was decreased to 13.41 by adding 600 ml of aqua DM.

Yield : Copper Zinc Ferrite ; Cux Zny Fe3-(x+y) O4,   Cux Zn(1-x) Fe2O4

Persamaan Reaksi ;   2 FeCl2.4H2O + 4 FeCl3.6H2O + 3x CuCl2.2H2O+ 3(1-x) ZnCl2 + 22 NaOH --> 3 CuxZn(1-x)Fe2 O4   + 22 NaCl + (42+6x) H2O

| Fraksi CuCl2,index 2x | | | | Fraks ZnCl2,index 2(1-x) | | | | Larutan Garam Klorida | | | | | | | | | | Larutan Basa | | | | | Volume |
| | | | | | | | | FeCl2.4H2O BM= 198.8 | | | | | FeCl3.6H2O BM= 270.33 | | | | | NaOH BM= 40 | | | | | Larutan Prekursor |
[Mol/lt]	[gram/lit]	gram/20	3x[gram/20	x	[Mol/lt]	gram/10m	3(1-x)gr/2	index reak	Mol/liter	gram/lit	gram/20m	gram/20m	index reak	Mol/liter	Mol/20m	gram/20m	gram/20m	index reak	Mol/litr	gram/llitr	[gram/20	pH + 800	[ml]
0	0	0	0	1	0.01	1.363	8.178	2	2	338	7.9532	7.953	4	4	0.08	21.626	21.626	22	22	880	17.6	13.73	37.758
0.2	34.08	0.6815	2.04456	0.8	0.008	1.0904	6.5424	2	2	338	7.9532	7.953	4	4	0.08	21.626	21.626	22	22	880	17.6	13.73	38.167
0.4	68.15	1.363	4.08312	0.6	0.006	0.8178	4.9068	2	2	338	7.9532	7.953	4	4	0.08	21.626	21.626	22	22	880	17.6	13.73	38.576
0.6	102.2	2.0446	6.13368	0.4	0.004	0.5452	3.2712	2	2	338	7.9532	7.953	4	4	0.08	21.626	21.626	22	22	880	17.6	13.73	38.984
0.8	136.3	2.7261	8.17824	0.2	0.002	0.2726	1.6356	2	2	338	7.9532	7.953	4	4	0.08	21.626	21.626	22	22	880	17.6	13.73	39.393
1	170.4	3.4076	10.2228	0	0	0	0	2	2	338	7.9532	7.953	4	4	0.08	21.626	21.626	22	22	880	17.6	13.73	39.802

NB; 1.Komposisi NaOH dalam tabel adalah komposisi ideal, agar seluruh garam klorida habis

berreaksi maka komposisi NaOH perlu ditambah.

   2. unttuk mencegah oksidasi, selain menggunakan aliran gas CO2, N2 atau Argon bisa juga digunakan

cara dengan menambahkan sedikit HCL . [Berger Patricia, Jonathan]

2.1 Preparation how to do the experiment as follows,

- 1 Clean the tubing coil, injector, PH electrode holder consecutively using sodium hydroxide solution, warm water, and run the nitrogen gas.

- Fill 2 injector input hose with DM aqua.

- Connect 2 injector input hoses with peristaltic pumps of sodium hydroxide solution and chloride salt solution

- Install pH datalogger and DeMux.

- Turn on DeMux, and Turn on (at record position) pH data logger

- Run peristaltic pump with discharge setting 30 cc / min

- At the beginning of the process there is still enough time to measure flow discharge, by draining the initial solution to the burette. Record alteration of surface surface of solution in buret with vide recorder.

- Immediately move the flow of the solution to the yield shelter.

- Hose a few seconds will look black liquid solution in the tube (tubing coil).

- Wait until the salt solution runs out. If you have exhausted the hoses to the place

- The black yield solution settles, then the washing process of the yield solution, samapai pH kl 7.

2.2 Qualitative Observation.

A) The process of reaction of FIS method successfully done with several obstacles, among others; Setting peristaltic pump speed and yield washing process.

B) Concentrations of concentrated saline solutions can lead to significant changes in the pH of the starting mixture of the process, resulting in different yield changes from the initial goal and the pH of the reactant in the reactor varied.

C) To anticipate the occurrence of a sharp yield difference, in addition to having to immediately adjust the pump speed but also disiasati with pH sodium hydroxide solution higher than the theoretical pH, so there is no sharp difference between the pH of saline solution (pH 1.8) with the pH of the basic solution (PH 13.

D) In order to obtain a relatively uniform yield solution after the reaction process, the starting solution settles in a brownish black color to be left about 1 hour after the reaction process to obtain a clear solution and brownish brown sediment.

E) Decreasing process depends on the pH of the yield solution, at pH 12 s / d 13 the average can precipitate rapidly (less than 1 hour). At the pH of the solution between 9 to 10 decansations takes more than 3 hours at pH below 9 dekatansi takes between 1 s / d 2 hours. [4]

F) The washing process using aqua DM is done after the perfect decanion process, but after pH 9 to 11 almost all samples show the same symptoms, which require a long time of settling yield. (A turbid solution is brownish, but the color of the solution changes as before blackening, then the yield settles but much longer than the post-reaction decanion) [4]

G) Washing yield required 5x to 7x decaption, for an average sample requires 12 hours washing time

H) The drying yield is carried out using a conventional small fire oven, requiring an average of 5 hours.

I) Sample Reaction Observation Table as follows

2.3 Tabel Observasi Reaksi Sampel:

Kode Sampel	$ZnCl_2$ [gram/20ml Aq]	$CuCl_2.2H_2O$ [gram/20ml Aq]	$FeCl_2.4H_2O$ [gram/20ml Aq]	$FeCl_3.6H_2O$ [gram/20ml Aq]	NaOH [gram/500ml Aq]	Temperatur reaksi	Metode FIS
TC X1.0P	0	10.22	7.95	21.6	>17.6	25	Circuler Hose Tube Coil
TCL X06P	3.27	6.13	7.95	21.6	>17.6	25	Circuler Hose Tube Coil
BL X06 P	3.27	6.13	7.95	21.6	>17.6	25	Circuler Hose Tube Coil
TC X04 P	4.90	4.09	7.95	21.6	>17.6	25	Circuler Hose Tube Coil

a) **Information.**

- Acid means the process is dominated by salt solution.
- Basa means process is dominated by alkaline solution
- Acid-base means in the process of changing the solution state.
- In the process should be dominated by Bases, but the Tube Coil FIS does not occur because in the process there is no mixing evenly (which caused no mixing process)

b) Process indicator, in addition can be done analytically with pH meters, but qualitatively and quickly can be done by looking at the yield color indicator on the reactor. The yield color on a normal process is brownish black.

- If the pH is less than 7 yellow color yield,
- If the pH is about 13 colors the yield is a brownish black and clumps of small clumps floating in a clear solution. Advanced Research

An immediate study will be conducted

1. Characterization of Yield Samples with XRD defrac- tors,

2. Characterization with Particle Size Analyzer (PSA)

3. Characterization with SEM or TEM depends on the PSA characterization results.

Sample Kode	pH Previous Solution, recording by pH SD Card	PH End Solution	Yield Solubility	Color of solution	Washing time And the lowest pH That can be achieved	Time Drying
TC X1.0P	Kl. 13.3	12.9	Settles	Brownish Black	>10 Hrs, pH 8.5	5hrs
TCL X06P	Kl.13.2	12.10	Settles	Dark chocolate	>10Hrs, pH 8.5	5 jam
BL X06 P	KL.13.35	13.35	Settles	Dark chocolate	>10Hrs, pH 8.6	5 jam
TC X04 P	KL.13.2	12.7	Relatively fast	Dark chocolate	>12 jam, pH 8.2	5 jam

2.4 Particle Size Analyser and Permagraph Test result:

CuFe2O4. 0.04 μm until 10μm. This particle has a size of 1 um is quite high, even those below 0.4 um about 48% volume. [5]
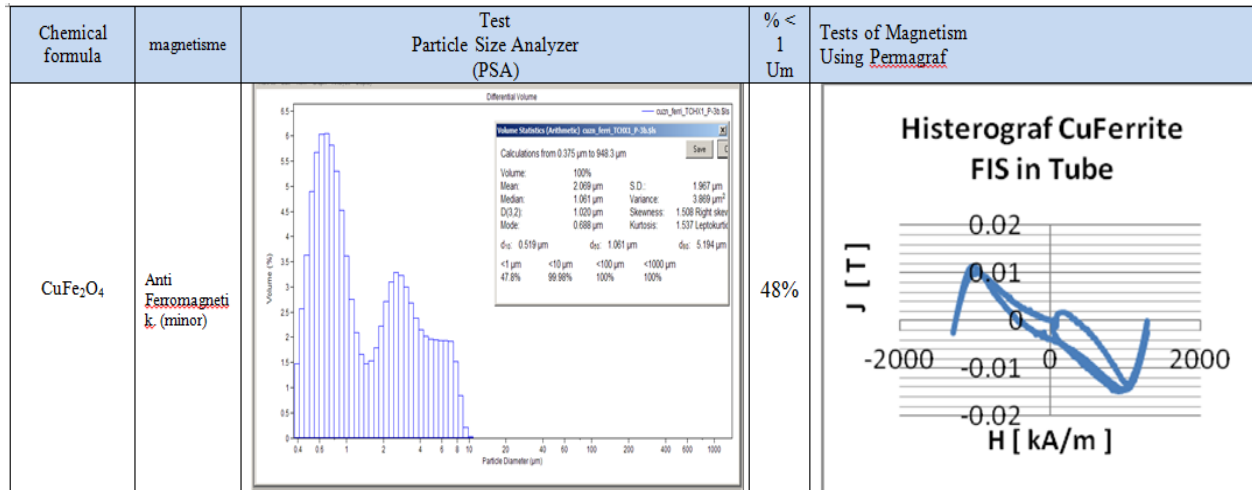
Chemical formula	magnetisme	Test Particle Size Analyzer (PSA)	% < 1 Um	Tests of Magnetism Using Permagraf
$CuFe_2O_4$	Anti Ferromagnetik. (minor)		48%	

Figure 3, $CuFe_2O_4$ This particle has a size of 1 um is quite high, even those below 0.4 um about 42% volume.

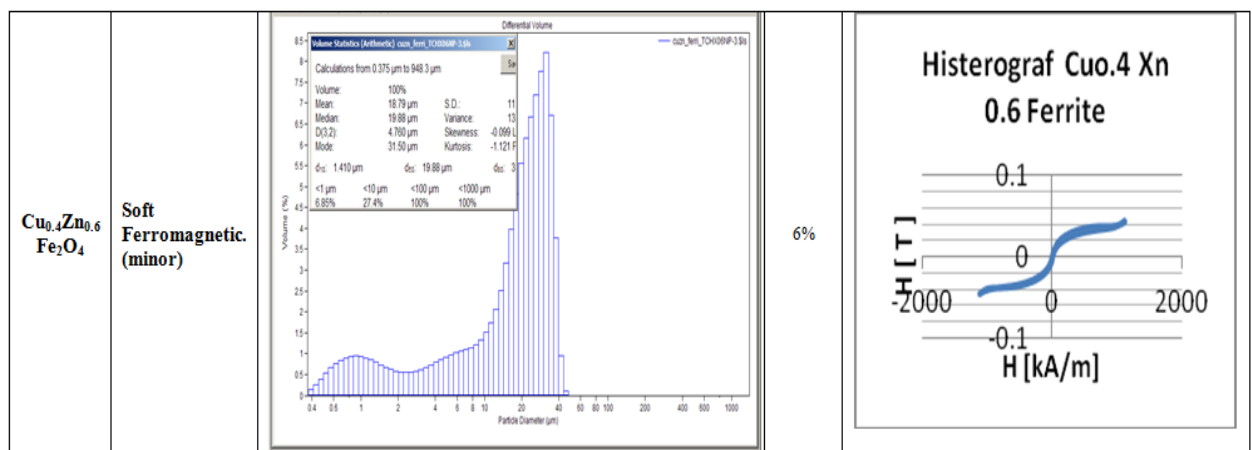|				
$Cu_{0.4}Zn_{0.6}Fe_2O_4$	Soft Ferromagnetic. (minor)		6%	

Figure 4, $Cu_{0.4}Zn_{0.6}Fe_2O_4$ These particles with size <1 um occupy about 7 % volume.
Particle size 0 to 10um is approximately 1.5% by volume, the rest of the size (10-40 um) occupies space 98.5% volume.
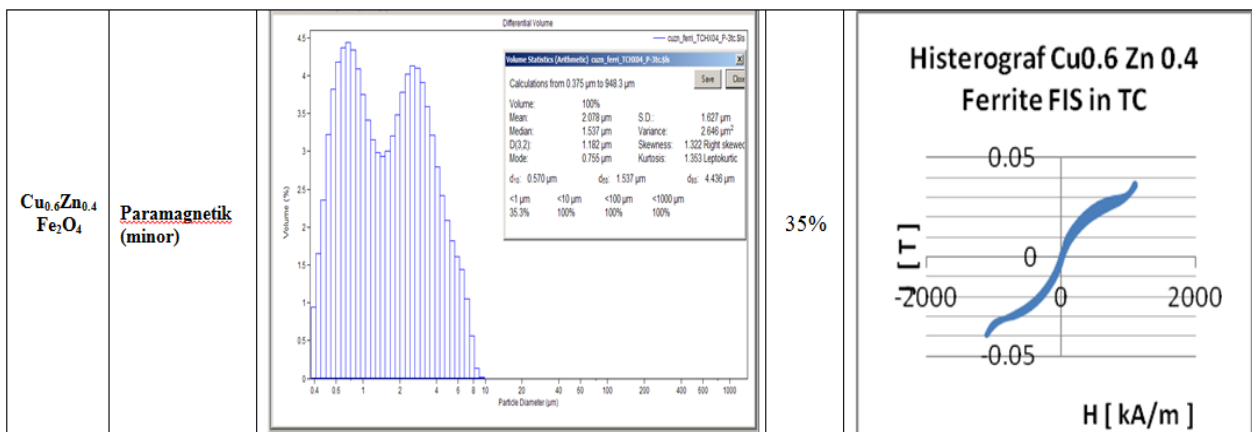
$Cu_{0.6}Zn_{0.4}Fe_2O_4$	Paramagnetik (minor)		35%	

Figure 5, $Cu_{0.6}Zn_{0.4}Fe_2O_4$ These particles with size <1 um occupy about 37% volume
In the mixing process between Cu0.4Zn0.6Fe2O4 and Cu0.6Zn0.4Fe2O4 is self-mutilated mixing, considering the two compounds have relatively different sizes, although from the same magnetism side is paraagnetic

3. RESULTS

Although from the same magnetism side is ferrom...

3.1 Particle Size Analyser And Permagraph:

Table Result of CuZnFerrite [5] Material Characterization Using PSA and Paragraph Qualitative Observation of Test Results of Characterization of PSA and Paragraph.

1. Non-linear particle size with the percentage of atom atoms of doping ferrite material.

2. The percentage of Cu and Zn doping atoms gives a sign of changes in the magnetism properties of the ferrite material as follows;

100% Cu doping, 0% Zn provides anti ferromagnetic properties but in a relatively sm (antiferromagnetic) J magnetic induction intensity.

80% Cu doping, 20% Zn gives soft ferromagnetic properties with significant magnetization intensity, Low relative energy level.

60% Cu 40% Zn provides soft ferromagnetic properties with smaller magnetization intensity than before 40% Cu doping Cu 60% Zn provides soft ferromagnetic properties with smaller magnetization intensity than before 20% Cu doping Cu 80% Zn provides minor anti ferromagnetic properties.

From point 2, it is possible to make diagram pattern of change of magnetic properties to the Percentage change of Cu and Zn atoms as doping atom of ferrite material as follows size of particle size on Cu 80% doping percentage gives percentage of nano size 80%, so material will Size of particle size on Cu 80% start having super paramagnetic properties (SPM) which is characteristic is same with ferromagnetic nature of material.

The moment in which the intensity of the magical field of zero and magnetic field indicates a certain value ie Br is a quantity called lately (remanensi). This Br is the magnetic remanence value remaining in the material after the magne field effect t dispensed. Where the strength of the magnet is determined by the magnitude of the Br value of the material. And the moment where the magnetic field is worth zero while the magnetic field intensity shows the value certain, Hc is called the coercivity force of ferromagnetic materials. This Hc value represents the magnitude of the reverse magnetic field required to remove the magnetism of a material.

For energy products (BHmaks) obtained from the multiplication of B and H, Where the higher the remanensi, the larger the 12 coercivity and the hysterical loop become fatter and the greater the energy product. A class of magnetic material often used to make

The permanent magnet is a ferrite material, which is an oxide composed by hematite (α-Fe2 O3) [5]

As the main component. In general ferrite is divided into three classes:

A. Soft Ferrite:

This ferrite has the formula MFe2O4 where M = Cu, Zn, Ni, Co, Fe, Mn, Mg [5,6,7] with crystal structure Such as spinel minerals. The nature of this material has a high permeability and resistance type, low coercivity.

B. Hard Ferrite:

This type of ferrite is a derivative of a plumbite magneto structure that can be written as MFe12O19, where M = Ba, Sr, Pb. This material has a high coercivity and remanence force and has a hexagonal crystal structure with magnetic moments parallel to the c axis.

This type of magnet is less expensive to produce and is widely used as a permanent magnet.

Ferrite Garnet Structured, this magnet has a spontaneous magnetization that depends on the temperature typically. The structure is very complicated, cubic shaped with unit cell compiled not me Ranging from 160 atoms [6].

From the hysteresis curve can be known the values of certain magnitudes namely the value of induction Remanen (Br), the value of the coefficient (Hc), maximum product energy value (BH max) and intrinsic coercivity of barium ferrite material (Hci).

In summary, the requirements for the material used as the magnetic core in high frequency inductors are:

-High permeability , -High saturation magnetization (4$\square$MSHigh uniaxial anisotropy field (Hk)

-Low coercivity (soft magnetic materials), -Sufficiently high resistivity to reduce eddy current losses   [1]

3.2 Ferrites:

Ferrites were Ferromagnetism material, which results in high permeability and high saturation, is prevalent in metals such as Co, Fe and Ni, they are not suitable for high-frequency[8] applications in bulk form because of their high conductivity leading to eddy current losses. Ferrites are based on oxides of these metals with higher electrical resistivity and thereby more suitable for power inductor applications. Spinel ferrites (e.g., NiFe2O4, MZn-and Ni-Zn-ferrites)[9] are extensively used in power converters because of their low losses than metal cores, resulting in high Q factors at moderate frequencies of 100 kHz to 1 MHz. However, for emerging high frequency consumer applications.

4. CONCLUSION

It has been successfully created and researched about the making of Flow Injection Synthesis Instrumentation by method of the Circuler Hose Tube Coil that work by chemical reaction test by drop in the semi-capillary pipe.   The raw material in the system was made by two kinds are salt and alkaline with presise calculation. Further measurements were made using Particle Size Analyzer for particle size, and Permagraph to determine the magnetism properties of the material.

In CuZnferrite making it appears that Cu and Zn atoms tend to break away. The Cu atoms are on the left while the Zn atoms are on the right Cu atoms carry anti ferromagnetic properties while Zn atoms carry ferromagnetic properties, one thing that can not be united. So if cu paired with Zn in the same will produce paramagnetic properties.

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