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Application of mixed colloidal magnetic fluid of single domain Fe_3O_4 and $NiFe_2O_4$ ferrite nanoparticles in audio speaker

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ABSTRACT

Ferrofluids are stable suspensions of colloidal ferrimagnetic particles in suitable non – magnetic carrier liquids. They have attracted a lot of attention from scientists and engineers due to their many interesting properties and applications in various branches of engineering. The present work reports the performance of colloidal fluid of single domain nanoparticles of $NiFe_2O_4$ and Fe_3O_4. The thermal properties and its dynamics on magnetization as well as its effect on thermal conductivity on the colloidal fluid are studied here. Advantages of the increased thermal conductivity and optimization of magnetization of mixed colloidal fluid is used to extract the heat from voice coil. Nanoparticles of 21 nm of Fe_3O_4 and 12 nm of $NiFe_2O_4$ are used for mixed colloidal fluid. The suspension of particles is achieved by coating the nanoparticles with mono-corboxylic group on both the types of particles. The higher size (21 nm of Fe_3O_4 and 12 nm of $NiFe_2O_4$) particles are taken for synthesizing colloidal fluid, to have magnetic property of mixed colloidal liquid at elevated temperature of voice coil of speaker (Higher sized particles gives better magnetization). Oil is used as a carrier. Mixed magnetic colloidal fluid is used as a medium for damping so that noise is reduced at higher temperature of voice coil.

Keywords: Audio speaker, nanoparticles of $NiFe_2O_4$ and Fe_3O_4, magnetization, mixed colloidal magnetic fluid.

I. INTRODUCTION

Nowadays, nanotechnology is emerging as one of the prime fields of technology. Nanotechnology deals with creation of useful nano-materials and devices through control of these matters on nanometer scale. Such a material has different phenomena and many physical properties that have been observed on this scale. The phenomena and benefits of this technology are used in making materials in many fields like nano-electronics [18, 20, 21, 22], optical fiber [1,17,19], computing, medicine and health, space exploration, environment and energy, aeronautics, magnetic recording media, magnetic fluid, catalysis, medical diagnostics, pigment in paint and ceramics [2-4] etc. Nano-science and technology is emerging as a very promising field as far as applications are concerned. Various Devices based on ferrofluids continue to offer high reliability and low cost solutions to many complicated industrial problems [5]. Many applications based on the susceptibility of the ferrofluid are devised [6]. Various studies on the magnetization of the magnetic colloidal fluid have been carried out [7]. Recently, extensive study on audio speakers containing ferrofluid has also been studied [8]. Their work was on increasing the radial restoring force on the voice coil and factors influencing splash loss of the ferrofluid due to shock. Ferrohydrodynamic analysis was employed for modeling. Ferrofluid is a temperature sensitive fluid, because they exhibit some properties like high thermal conductivity, small change in temperature, large change in magnetization. In case of mixed colloidal ferrofluids, small change in temperature and small change in magnetization occurs. This work deals with the preparation, characterization and application of the mixed ferrofluid on audio speaker for performance up gradation and optimizing the magnetization of mixed colloidal fluid. We aim at designing of colloidal magnetic fluid for consistent performance on heat extraction, noise damping and high fidelity achievement with minimum deviation in magnetization against temperature. The $NiFe_2O_4$ have been used because of its high permeability in higher frequency range and low core losses at such frequencies, as well as somewhat flat magnetization characteristics in the temperature span of interest.

The heat generated by the voice coil will be partly transferred to speaker components by the way of convection and conduction under the situation when only air is present in the coil. This process is not so efficient. The pioneering work on application of ferrofluid in speaker was carried out with reference to centering of voice coil and enhancement in performance of voice coil with ferrofluid in the vicinity of the voice coil [9].

II. PREPARATION OF $NiFe_2O_4$ AND Fe_3O_4 NANOPARTICLES AND THEIR FERROFLUIDS

The particles are prepared by chemical co-precipitation method and dispersed in the carrier liquid with stabilizer. The nanoparticles are prepared using aqueous solution of $FeCl_3$ and $NiCl_2$ with (2:1) proportion in ammonium hydroxide to achieve $NiFe_2O_4$ magnetic nanoparticles. Ferritization of NiO and Fe_2O_3 was achieved at 98 ^{0}C. Oleic acid molecules were adsorbed on particles. Particles were decanted and washed with acetone as well as warm water three times. Collected particles were dried in oven and dispersed in transformer oil as carrier. Dried powder sample was preserved for XRD characterization. Ferrofluid was centrifuged at 4500 RPM for one and half hour. Lumps were removed and clean solution was obtained. The volume fraction Φ_m of 0.08 was prepared for $NiFe_2O_4$.

Similarly, Fe_3O_4 magnetic nanoparticles were also prepared with chemical co-precipitation method. Aqueous solution of salts of $FeCl_2$ and $FeCl_3$ with 1:2 proportions in NaOH base solutions were used to achieve Fe_3O_4 magnetic nanoparticles. The ferritization was readily achieved at room temperature without any difficulty. Surfactant molecules of Oleic acid were adsorbed on particles. Particles were decanted and washed. Magnetite powder was dried in oven for 24Hrs at 80^{O}C. Powder was dispersed in transformer oil. Samples were centrifuged at 4500 RPM for one and half hour. Lumps were removed and clean ferrofluid was obtained. Dried sample was also preserved for XRD characterization. Ferrofluid with volume concentration of 0.08 was prepared.

III. CHARACTERIZATION OF $NiFe_2O_4$ AND Fe_3O_4 NANOPARTICLES

TEM of the sample of $NiFe_2O_4$ was obtained as in Fig 1(a). The morphology and particle size of this nanoparticle was characterized by transmission electron microscopy (TEM, H-7650 accelerating voltage of 120 KV).TEM shows the particle size of about 12 nm.

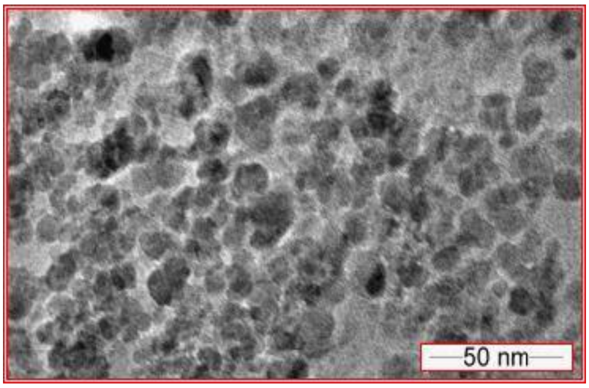


Figure 1(a). TEM image of $NiFe_2O_4$ ferrofluid sample.

X-rays diffraction characterization has also been performed on the Nickel substituted particles. XRD characterization was carried out by using powder X-ray diffractometer (Phillips PW 1800, range is 6-80^O 2θ). Fig.1(b) shows that the particles have inverse spinel structure of calculated size of 12.3 nm. The details of plane d- spacing and 2 theta values are given in table 1.

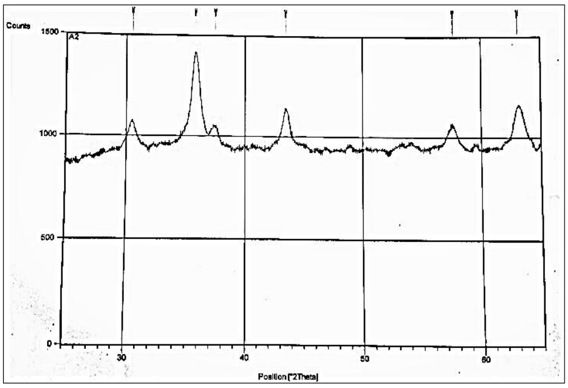
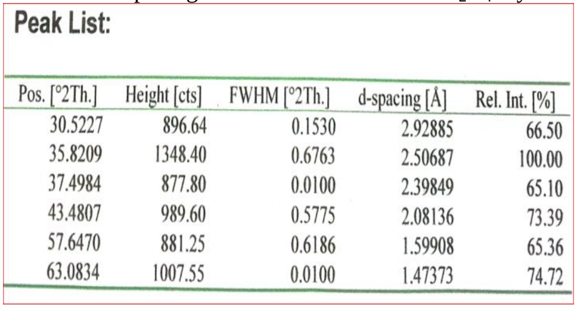


Figure 1(b). XRD pattern of $NiFe_2O_4$ powder.

Table 1. d-spacing and 2theta values for $NiFe_2O_4$ crystal.



Peak List:

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
30.5227	896.64	0.1530	2.92885	66.50
35.8209	1348.40	0.6763	2.50687	100.00
37.4984	877.80	0.0100	2.39849	65.10
43.4807	989.60	0.5775	2.08136	73.39
57.6470	881.25	0.6186	1.59908	65.36
63.0834	1007.55	0.0100	1.47373	74.72

TEM of the sample of Fe_3O_4 was obtained as in Fig 2(a). TEM shows the particle size of about 21 nm.

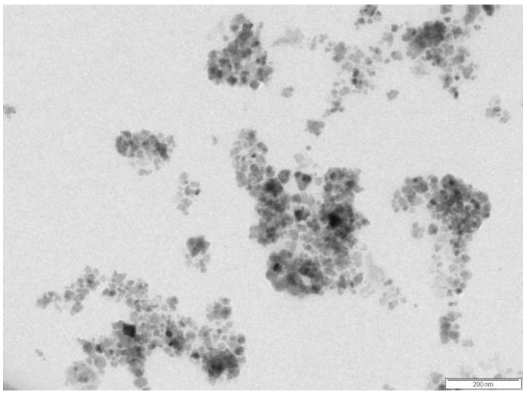


Figure 2(a). TEM of Fe_3O_4 system with 21 nm particle size ferrofluid.

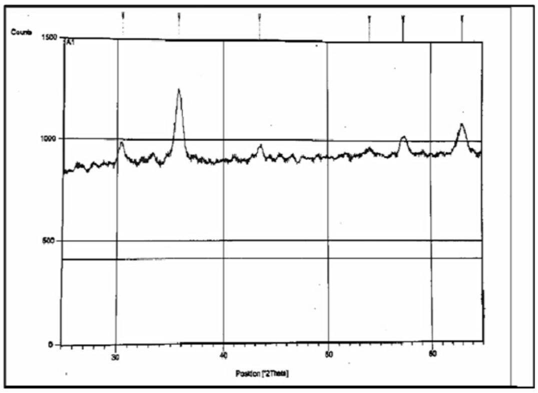
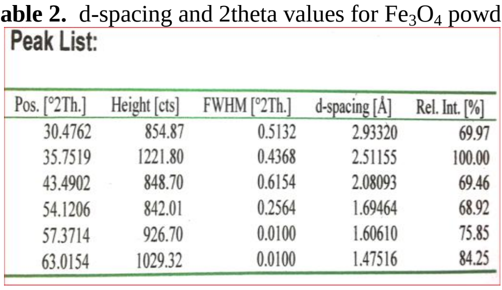


Figure 2(b). XRD pattern of Fe_3O_4 powder**.**

Table 2. d-spacing and 2theta values for Fe_3O_4 powder



Peak List:

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
30.4762	854.87	0.5132	2.93320	69.97
35.7519	1221.80	0.4368	2.51155	100.00
43.4902	848.70	0.6154	2.08093	69.46
54.1206	842.01	0.2564	1.69464	68.92
57.3714	926.70	0.0100	1.60610	75.85
63.0154	1029.32	0.0100	1.47516	84.25

The structural properties of produced nanoparticles were analyzed by XRD taken on Philipsmake X'pert PRO model XRD machine as shown in Fig 2(b). XRD wavelength □of 0.15406nm was used for characterization.

$D = K\lambda/ (B\cos\theta)$

B is a full width half maximum (FWHM), K is the shape parameter and 2θ is the bragg's angle. The value of K is 0.89 for magnetite. The largest intensity in the graph is at [311] plane with 2θ of 35.7^o. Half maximum intensity width of the peak accounted with instrument broadening; particle size was calculated to be 19 nm by Scherrer's formula.

Particles prepared are single domain particles, as such can be considered in a state of uniform magnetization with a magnetic moment *m*. $m = M_dV$, where M_d is the domain magnetization, V is the volume of particle. The mean magnetic moment *m* of a particle in the field direction is described by Langevin expression.

$m = M_d [\coth \xi – 1/\xi]$

Where,

$\xi = (mH_o/ kT)$.

The behavior of the ferrofluid depends upon the behavior of individual particle. The Brownian relaxation time $\tau_{B =}$ 3 Vη / kT, Neel relaxation time $\tau_N = \tau_O\ e^{\sigma}$ affects the susceptibility of the particle, where V is the hydrodynamic volume of a particle, η is the dynamic viscosity of the carrier liquid, σ is associated with anisotropy of the crystal K and given with KV / kT and τ_O, the damping or extinction time is about $\sim10^{-9}$ seconds. In Neel relaxation, the magnetic moment may change or reverse the direction within the particle without the rotation of the particle. This is achieved by overcoming the energy barrier. The probability of such a transition is approximately equal to e^{σ}. It is interesting that when $\tau_N = \tau_B$, one can determine critical particle radius; above this radius, the Brownian relaxation mechanism occurs and below this, is the relaxation by Neel's mechanism. The total susceptibility of the ferrofluid depends on number of particles contribution. The X_i can be shown as a function of volumetric fraction Φ_m. Where $X_i = (\pi/18)\ (\Phi_m)\ (\mu_0 M_d^2 d^3/kT)$. The mean particle size was d≈21 nm in this work. The volume concentration Φ_t of the particles coated with oleic acid in the carrier has Φ_t=0.08. The carrier used was transformer oil for its stability at higher temperature.

IV. MAGNETIZATION OF MAGNETIC NANOPARTICLES

Magnetization of nanoparticles of magnetite is studied under applied transient field at various packing fractions [11]. Field induced particle alignment and magnetization of ferrofluid was also studied [12]. Magnetization of particles was deliberated that the shell of the particle affects magnetization of the particle [13]. However classically, a mathematical modeling on magnetization of particle is carried out and magnetic saturation of particle [14] is given by equation 1.

$$M_{sat} = mM_d(r-d)^3/r^3 \qquad (1)$$

m is mass of the particle,
M_{sat} is Saturation magnetization of the particle,
M_d is Domain magnetization of material,
d is shell thickness of particle,
r is radius of the particle. Similarly,

Spontaneous magnetization which exists in the absence of external field H can be given by equation 2 depends upon saturation magnetization of particle.

$$M_{spo} = M_{sat}\ \{[1-0.3(T/T_c)^{1.2}]\} \qquad (2)$$

Where, T is temperature, T_c is Curie temperature, M_{sat} is saturation magnetization of particle and M_{spo} is spontaneous magnetization.

$$M= M_{sat}\ [\ \coth (m_p\ H/kT) –(kT/\ m_p\ H)]+ X.H \qquad (3)$$

m_p can be calculated from equation 4.

$$M_{sat} V = n.m_p \quad (4)$$

Whereas, this formula is for system of particles; where, m_p is a magnetic moment of a particle. X is the susceptibility of the particle; H is auxiliary applied magnetic field [15]. The susceptibility X of a nanoparticle is proportional to its volume V, Spontaneous magnetization M_{spo} and inverse of temperature T.

$$X = Mspo^2(\mu_o V/3k_bT) \quad (5)$$

Where μ_o is the permeability of free space, k_b is Boltzmann's constant, T is the temperature, M_{spo} is Spontaneous magnetization. The entire description of any magnetic particles susceptibility for various cases is quite complex. It involves computations of imaginary parts of the susceptibility term that arise below 150K and complex non-linear behavior for the particle in a dynamic field (Jonsson et al. 2000). The following values of susceptibility are valid for temperatures above 150K and carried out at room temperature. The coerceivity of clusters of particles are important due to their coupling effects as well as dependency on volumetric concentrations [16].

V. FERRITE NANOMATERIALS

M-H curve of such nanomaterials are generally super-paramagnetic in nature and do not show hysteresis or show very low loss in applications. In addition to this, one property such as electrical resistivity of such materials is also very high.

First choice for such materials may be $Fe^{2+}[Fe_2^{3+}]O_4$ which is magnetite and easily prepared at room temperature. Such types of composition is $M^{2+}[Fe_2^{3+}]O_4$ where M can be Mn^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+} or Fe^{2+} ions. Ferrite materials are spinel materials. There are generally two types of spinel materials. First type is simple spinel and other type is mixed or random spinel. Simple spinel ferrite materials are further classified as Normal spinel and Inverse spinel materials. In normal spinel structure such as $(M^{2+})[Fe_2^{3+}]O_4$; M^{2+} resides at Tetrahedral site and Fe_2^{3+} resides at Octahedral site. Parenthesis ( ) is for tetrahedral site and bracket [ ] are for octahedral site. Where as in inverse spinel structure such as $(Fe^{3+})[M^{2+}.Fe^{3+}]O_4$, M^{2+} reside at Octahedral site and half of Fe_2^{3+} are distributed at Tetrahedral and Octahedral sites respectively. Trivalent parts of octahedral and tetrahedral are anti parallel to each other. The magnetic moment of M^{2+} and Fe^{3+} ions in octahedral sites are aligned in same direction. Most of the spinel ferrites form cubic spinel structure with oxygen anions in FCC positions and cations in the tetrahedral and octahedral coordinated interstitial lattice sites. This will form two sub lattices, octahedral and tetrahedral. The inter-sublattice interactions i.e. JAB: A-O-B is extremely stronger than the intra-sublattice interactions JAA: A-O-A and JBB: B-O-B in spinel ferrites with collinear ferrimagnetic structure. Extensive works on certain spinel ferrites have been carried out in the last few decades, because of their theoretical understanding and potential applications in science and technology. In $(Fe^{3+})[M^{2+}.Fe^{3+}]O_4$ as inverse spinel, the magnetic moments for Fe^{2+} is $4\boldsymbol{\mu}_B$ and Fe^{3+} is $5\boldsymbol{\mu}_B$. Fe^{3+} , while in tetrahedral site is anti-ferromagnetically coupled with Fe^{2+} and Fe^{3+} in octahedral site reveals a net magnetic moment of $4\boldsymbol{\mu}_B$ for Fe_3O_4 molecule.

In mixed spinel structure such as $(M^{2+}_{1-x}Fe^{3+}_{x})[M^{2+}_{x}.Fe^{3+}_{2-x}]O_4$, M^{2+} and Fe^{3+} partially resides at Octahedral site and tetrahedral sites respectively. If divalent ions are in octahedral and tetrahedral sites, then structure is disordered. On x=0, the structure reduces to normal spinel. X=1 will reduce the structure to Inverse spinel. X=2/3 would make random structure or distributions of cations [10].

The relative positions of divalent ions and trivalent ions to neighboring oxygen ion are very important for strong super-exchange interaction. This is dependent on cation-anion-cation bond angle as well as inter-atomic spacing between cations and anions.

In unit cell, there fits eight molecules of Fe_3O_4; Oxygen molecules are tetrahedrally coordinated but metal ions are tetrahedrally and octahedrally interstice between oxygen.

VI. SPEAKER CONSTRUCTION AND COMPONENTS

A speaker is a Transducer that converts electrical signals to mechanical vibration. This section explains the construction and fundamental principles of working of a speaker. There are many components and the overall performance of the system mainly depends up on the performance of the voice coil among those components. The frequency band of the interest is from 20Hz to 20000Hz. Ideally the output of the speaker should be proportional to the signal applied, but this is not what is observed. The power of the speaker also called the sound pressure depends on various factors; one of those factors being the characteristics of the signal itself. Frequency response of the signal emitted from speaker to the given input of the speaker is the main characteristic. In other tests the response of the speaker is evaluated from 1 meter from the test speaker. In this work, this technique is used to evaluate the frequency response of the speaker with and without ferrofluid insertion in the voice coil gap. High fidelity is another characteristic of speaker which is a measure of precise production of sound according to the electrical input. Precision parameters deal with an ability of speaker to reproduce the same signal repeatedly with deviation within prescribed limit. There are different components in a speaker as per Fig 5(a), Fig 5(b). Fig 5(a) shows the components of speaker in the order it gets retrofitted. From the Top in Fig 5(a) is dust cap, diaphragm, spider, voice coil, speaker frame and magnetic assembly. Fig 3 shows frame and the gap for voice coil where the ferrofluid is inserted. Refer to Fig.3 where the center of the gap ring is a South Pole where the gap ring is surrounded by magnet and North Pole at the top of the magnet. The diaphragm is connected to frame with the suspension ring at the upper side. Figure 4 shows the functional diagram of speaker with inserted ferrofluid along the voice coil.

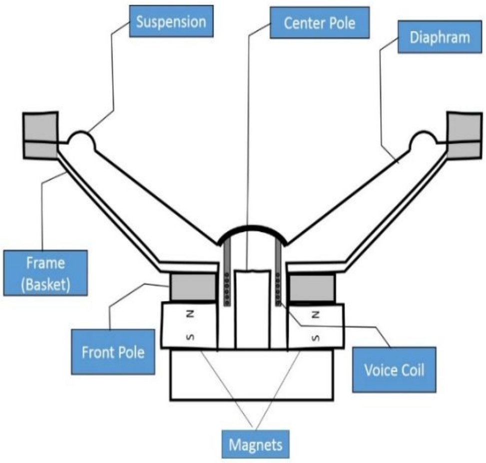


Figure 3. Speaker components.

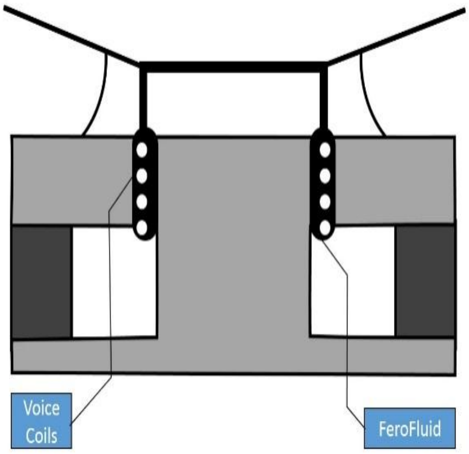


Figure 4. Showing ferrofluid functional with voice coil.

Figure 5(a). Speaker Components

Figure 5(b). Speaker components.

In brief, speaker consists of two main parts, the electro-mechanical part and the mechanical-acoustic part. The Electro-Mechanical part consists of a coil and a magnet; the arrangement is such that the coil is concentric to the center pole of the magnet. The shape of the magnet creates a radially uniform field. When the current flows through the coil, it produces a magnetic field. The strength and direction of the field depends on the direction (i.e. clockwise or anticlockwise) and magnitude of the current. The coil is attached to the diaphragm using proper adhesive and hence when the coil moves it forces the diaphragm to move with it.

The Mechanical-Acoustic motion of the coil, moves the diaphragm too, the vibration of which creates pressure waves. In order to improve quality of sound, it is ideally preferred that the diaphragm be perfectly rigid and light in weight. But since no material is perfectly rigid, the cone is purposely designed to flex. The cone has to be rigid enough to produce quality sound over maximum range of frequency yet light enough to transfer most of power from electrical form to pressure waves. If the natural frequency of the cone lies within the audio frequency range or if the vibrations generate harmonics, this could affect the performance of the diaphragm. Usually materials with low mass high density, good temperature stability and selective damping property are selected for making the cone.

Efficiency of speaker depends on power losses and these two parts contribute differently to these losses. While most of the power loss occurs in the mechanical-acoustic part i.e. diaphragm, there is a certain loss through the voice coils in the electro-mechanical part in the form of Ohmic loss or heating. The losses due to diaphragm can be controlled using proper speaker size and right material for the diaphragm. The current from the voice coil heats itself up, thereby increasing its resistance and dissipating energy, this heat also expands the coil causing damage to the coil diaphragm attachment and thus reducing the speaker's life. It is therefore necessary to conduct the heat away. It is observed that injecting ferrofluids into the gaps of the coil increases the life of the coil significantly. This is because the ferrofluid conducts the heat away from the coil to the pole of the magnet. Also, the presence of the ferrofluid helps keep the coil practically concentric all the time thus increasing the fidelity of the sound due to levitation effect of a ferrofluid on the electromagnet.

Another problem faced by voice coil is the non-linearity in the magnetization of ferrofluid with respect to its temperature. This problem is less addressed elsewhere; the present work addresses this problem by fine tuning the system, so that the non-linearity in magnetization is minimum.

VII. EXPERIMENTAL SETUP

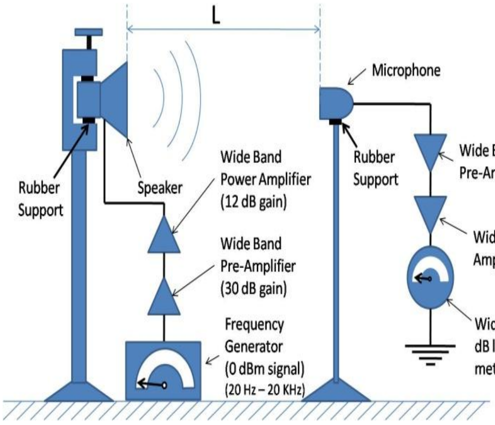


Figure 6. Experimental setup for speaker and microphone to study frequency response.

Experimental setup as shown in Fig. 6 is used for taking readings for finding out the frequency response of the system.

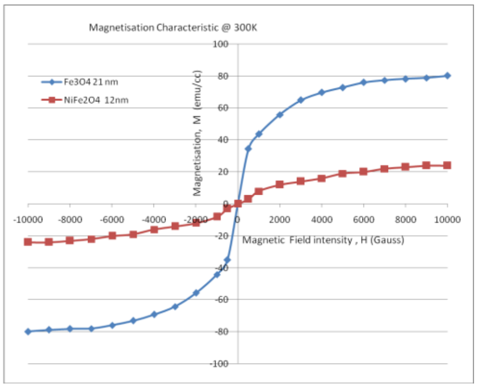


Figure 7. VSM data for Fe_3O_4 21nm, $NiFe_2O_4$ 12nm nanoparticles.

Table 3. Showing magnetization @300K (at $H=10^3$ Gauss) and % non-linearity in the range of 300K to 475K operating temperature range of speaker

Sample	% of $NiFe_2O_4$	% of Fe_3O_4	M_i(sat) emu/cc	% Non-linearity
A	10	90	3.75	0.475
B	20	80	3.5	0.455
C	30	70	3.2	0.410
D	40	60	3.0	0.350
E	50	50	2.75	0.275

VSM data of solid phase of $NiFe_2O_4$ and Fe_3O_4 is shown in Fig 7. The saturation magnetization of about 20 emu/cc and 80 emu/cc has been observed for $NiFe_2O_4$ and Fe_3O_4 nanoparticles at magnetic field intensity of 1 Tesla. These particles have been used for preparation of mixed ferrofluid.The magnetization of mixed ferrofluid is done by standard Quinck's method. Table 3 shows the variation in magnetization of such mixtures with temperature.

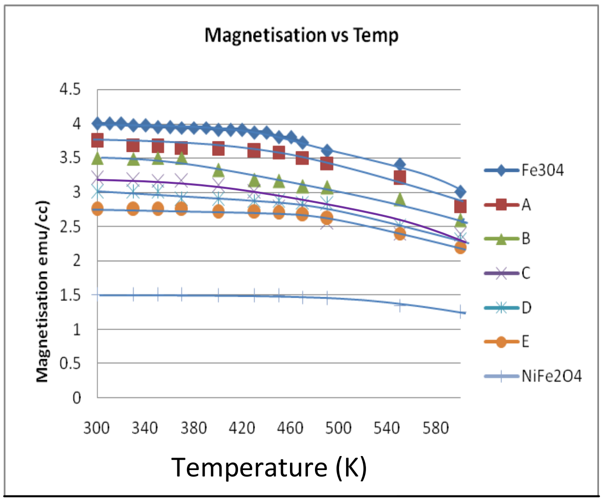


Figure 8. Sample A, B, C, D, and E with control samples showing variation of magnetization with temperature.

Magnetization variation of mixed ferrofluid with temperature is as shown in Fig. 8. We conclude that as the temperature increases, magnetization is nearly steady up to the room temperature. Above room temperature, magnetization decreases with increase in temperature. Higher non-linearity is observed in pure Fe_3O_4 as compared to sample A,B,C,D,E. Nonlinearity decreases in sample A,B,C,D,E due to mixed colloidal magnetic fluid.

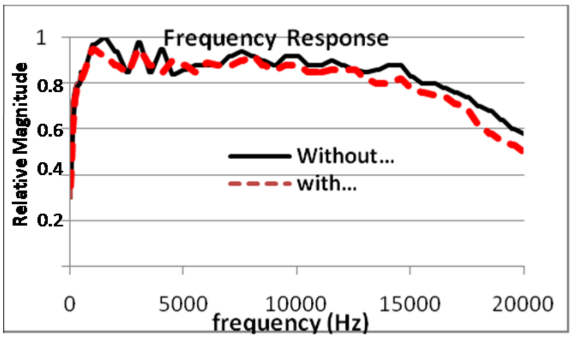


Figure 9. Frequency response @ 30dBm signal at speaker input and detection at 1 meter.

VIII. RESULTS AND DISCUSSIONS

It has been observed that the thermal conductivity decreases with rise in temperature in transformer oil as shown in Fig. 10. Thermal conductivity increases with increase in volume concentration. As temperature increases, Brownian motion increases and aggregation time decreases. This leads to higher probability for particles aggregation. Whereas, the aggregation of particle is more prominent and is governed by surface chemistry rather than thermal agitation. The particles are coated with surfactant and stabilized for greater than at least 10kT, to prevent aggregation due to various factors including thermal agitation.

Another important fact observed was reduction in viscosity with the rise in temperature as in Fig. 11. The dependence of viscosity on the temperature can be given by Arrhenius expression.

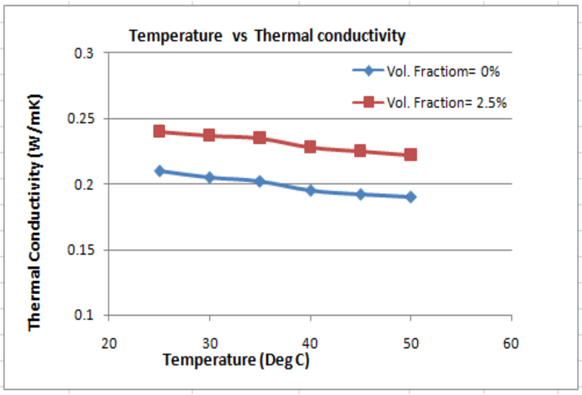


Figure 10. Temperature vs Thermal conductivity

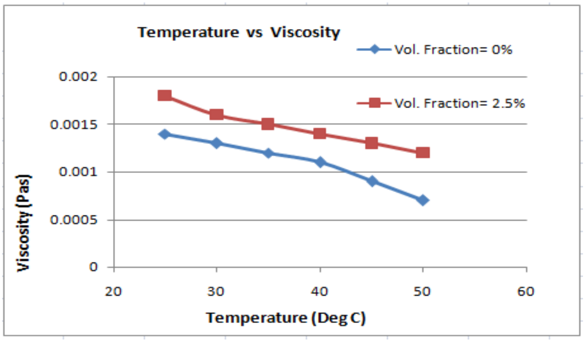


Figure 11. Temperature vs viscosity

The ferrofluid plays three important roles when inserted in the gap of voice coil and magnet of loudspeaker. It conducts heat away from the voice coil, which increases the power handling capacity. It keeps the voice coil concentric with the core magnet so that high fidelity is achieved and high frequency noise is reduced. Sample E has been observed to give least non-linearity with variation in temperature.

IX. CONCLUSIONS

It is observed that mixed ferrofluid of $NiFe_2O_4$ and Fe_3O_4 with 1:1 ratio gives least nonlinearity against temperature. An improvement in high frequency response is observed. Reducing the amount of Fe_3O_4 below 50% would further reduce the magnetization of the mixture and would badly affect on the performance of speaker, as it is less efficient to maintain centering of voice coil and damping noise. Mixed magnetic colloidal fluid is used as a medium for damping so that noise is reduced at higher temperature of voice coil.

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