

# Effect of NiO Filler on the Dielectric Constant and morphology of ZnO using Solid State Method at Microwave Frequency

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**Abstract-** Dielectric materials have many important functions in the microelectronics industry. The aim of this research is to characterize the dielectric constant of doped zinc-oxide composites using solid state method at microwave frequency. The methods used in this research are solid state method for sample preparation, open ended coaxial probe (OECF) for determining the dielectric constant and FTIR for bonding and IR absorption properties. The OECF results shows a sequential increase in dielectric constant as pure ZnO is doped incrementally with the filler (NiO). It also shows a sequential decrease in dielectric constant as the frequency increases. The FTIR result shows an increase in IR absorption as NiO content increases. The result from SEM was able to distinguish between the filler and matrix for each composition. Therefore NiO can be used as a filler for improving dielectric constant of ZnO as a matrix. The composite can also serve as a good agent for constructing capacitors and other dielectric materials which are hence used in manufacturing electronic and telecommunication gadgets. It was also proven that solid state method is a good method for synthesis of powdered sample like ZnO and NiO for determining their dielectric constant.

**Key Words -** Nickel Oxide (NiO); Zinc Oxide (ZnO); Dielectric Constant; Solid State Method; Microwave Frequency

## I. INTRODUCTION

Mixed oxide nanocomposites are being studied because of their potential for enhanced functional performance in photocatalysis, sensors and other optoelectronic device applications. ZnO is a wide band gap (3.2 eV) n-type semiconductor that has found a wide range of applications as a transparent conducting oxide (TCO) electrode in photovoltaics, photocatalysis, sensing, fuel cells and other optoelectronic devices (Albert & Abiola, 2017). Umit, (2010), reported that, ZnO is an attractive material for applications in electronics, photonics, acoustics, and sensing. In optical emitters, its high exciton binding energy (60 meV) gives ZnO an edge over other semiconductors such as GaN if reproducible and reliable p-type doping in ZnO were to be achieved, which currently remains to be the main obstacle for realization of bipolar devices. On the electronic side, ZnO holds some potential in transparent thin

film transistors (TFTs) owing to its high optical transmittivity and high conductivity.

NiO is a p-type wide band gap (4.2 eV) semiconductor that has been used in similar applications. ZnO and NiO readily form a p-n junction that has shown good electrical properties for gas sensing, fuel cell electrodes and photocatalysis (Albert and Abiola, 2017). The applications of Nickel oxide (NiO) today is found in semiconductors, capacitor-inductor devices, tuned circuits, transparent heat mirrors, thermistors and varistors, batteries, micro-supercapacitors, electrochromic and chemical or temperature sensing devices. It is used in preparation of nickel cermet, plastics and textiles, in nanowires, nanofibers and specific alloy and catalyst applications. It is also used as an antiferromagnetic layers, accelerators and radar absorbing materials, aerospace and active optical filters (Sani *et al.*, 2019; AzoNano, 2013). Ghassan and Naeem (2016), in their research investigated the effect of volume filler content  $\phi$  on dielectric constant of polyethylene PE filled with nickel (Ni) powders. They observed that, the dielectric constant of such composites increase suddenly at a critical volume concentration indicating a semi-conducting behaviour. Aldar *et al.* (2014), reported that the dielectric constant decreases with increase in frequency. The dielectric constant decreases rapidly at low frequencies and becomes quite slow at high frequencies. The higher value of dielectric constant at lower frequencies is explained on the basis of space charge polarization. Ranga *et al.* (1999), investigated the dielectric constant of polycrystalline mixed nickel-zinc ferrites in the frequency range 100 kHz–1 MHz. It was shown that, the dielectric constant for these ferrites is approximately inversely proportional to the square root of the resistivity. John *et al.* (2008), prepared nanostructured nickel ferrite samples through a chemical precipitation method followed by thermal processes. The dielectric constant was studied and the nanoparticles of nickel ferrite showed substantial variation in the values of dielectric constants.

A series of nanocomposites of nickel-zinc ferrite + paraformaldehyde was successfully synthesized using the mechanical milling process. With the increase in the volume of polymer, the dielectric constants of all the composites

decreases (Raju & Murphy, 2012). Nickel nanocomposites were synthesized by thermal decomposition route. The dielectric constant, was observed to decrease rapidly with increase in frequency in the low frequency range and it reach a constant value at the high frequency range which is independent of frequency (Sourav *et al.*, 2014). The composite of oil palm empty fruit bunch fiber (OPEFB) which is the waste product of oil palm industry, environmentally friendly polycaprolactone (PCL) and nickel oxide (NiO) were fabricated by compounding all materials in the Thermo Haake blending machine. The dielectric constants of the substrates were obtained with the open ended coaxial method for microwave frequency range between 0.2 MHz and 20 GHz. The results revealed that the permittivity values of the composite can be tuned by changing the ratio of OPEFB/PCL/NiO prior to compounding and blending (Ahmad *et al.*, 2015). Gaurav *et al.* (2015), studied the improvement in dielectric constant of nematic liquid crystal (NLC) by doping of nickel oxide (NiO) nanoparticles. They observed that the dielectric constant decrease as the frequency increases. Ajai *et al.* (2017), synthesized conducting polyaniline-nickel ferrite (PANI/NiFe<sub>2</sub>O<sub>4</sub>) composites by employing interfacial polymerization method. The dielectric constant of PANI/NiFe<sub>2</sub>O<sub>4</sub> composites are large at lower frequencies and decrease steeply with increasing frequency. Ramesh *et al.* (2003), measured ultralow dielectric constant values on Ni-Zn ferrites prepared using Fe<sub>2</sub>O<sub>3</sub> as a starting material and sintered in a microwave field. Dielectric properties were observed between microwave-sintered Ni-Zn ferrites prepared using Fe<sub>3</sub>O<sub>4</sub> (T34) and those starting with Fe<sub>2</sub>O<sub>3</sub> (T23) ingredients. The ultralow dielectric constant values observed on T23 ferrites show that this procedure is highly suitable to prepare Ni-Zn ferrites for high-frequency switching applications. Bahari *et al.* (2012), fabricated polyvinylpyrrolidone / Nickel oxide (PVP/NiO) dielectrics with sol-gel method using 0.2 g of PVP at different working temperatures of 80, 150 and 200 °C. The obtained results demonstrate the feasibility of using high dielectric constant nanocomposite PVP/NiO as gate dielectric insulator in the organic thin film transistors (OTFTs). Banerjee *et al.* (2012), synthesized Barium strontium titanate (BST) ceramics (Ba<sub>0.6</sub>Sr<sub>0.4</sub>)TiO<sub>3</sub> by solid state sintering using barium carbonate, strontium carbonate and rutile as the precursor materials. The samples were doped with nickel oxide in different proportions. It was observed that the dielectric properties of BST were modified significantly with nickel oxide doping. Muhammad *et al.* (2012), focus on the preparation of nickel oxide nanocrystallites by a novel low cost sol-gel auto-combustion technique and the dielectric constant was studied. The value of dielectric constant decreased with the increase of frequency up to 1 MHz, which they attributed to the space charge polarization. Rajashekhar *et al.* (2015), synthesized NiO nanoparticles by self-propagating high temperature (SHS) method. These synthesized NNP are used to blend with PANI to get PANI -NiO (PN) composites. The result of the dielectric behaviour of the above synthesized nanocomposites suites for battery applications.

The FTIR spectra for pure PVA, ZnO/ PVA nanocomposite films and ZnO are reported by Mansour, *et al.* (2014). In the spectra of pure PVA and ZnO/ PVA nanocomposites, the broad and strong band centered at 3340 cm<sup>-1</sup> is assigned to the stretching vibration of hydroxyl group (OH). The strong band at 2940 cm<sup>-1</sup> is assigned to the band of asymmetric CH<sub>2</sub> stretching. The two bands observed at 1712 and 1658 cm<sup>-1</sup> are assigned to the stretching vibrational band of C=O. The two bands observed at 1427 and 1330 cm<sup>-1</sup> are assigned as CH<sub>3</sub> bending vibration and CH<sub>2</sub> stretching respectively. The band at 1090 cm<sup>-1</sup> arises from the C-O stretching vibration while the band at 920 cm<sup>-1</sup> results from CH<sub>2</sub> rocking vibration. Also, the band at 850 cm<sup>-1</sup> result from C-C stretching vibration and that at 660 cm<sup>-1</sup> arises from out of plane OH bending. The band at 432cm<sup>-1</sup> is assigned to the stretching vibration of Zn-O bond. FTIR spectra of PVA, ZnO and ZnO/ PVA nanocomposites indicating that there are no interactions between polyvinyl alcohol and zinc oxide in forming nanocomposites.

Nisha *et al.* (2008), prepared nanoparticles of nickel-cobalt oxide by chemical co-precipitation method. The effect of frequency on the dielectric behaviour have been studied for nanosized samples of nickel-cobalt spinel oxide. It is seen that, with the decreasing frequency, the dielectric constant increases much more obviously than that of the conventional materials. Osama *et al.* (2014), reported that, addition of SiO<sub>2</sub> in the presence of ZnO and NiO leads to better densification by minimizing the present of closed pores. Results of firing shrinkage as a function of temperature show increase of shrinkage with temperature. The dielectric constant shows a decreasing trend for all the samples, the decrease is rapid at lower frequency and slower and stable at higher frequency.

Latif *et al.* (2012), investigated the dielectric behavior of the polar (CPVA) /ZnO nanocomposite films. The results show that the dopant composition has great influence on the magnitude of dielectric properties. The results also show that the composite polymer films have both electric and electronic properties. Wang *et al.* (2017), prepared the samples of 1%, 2%, 3% and 4% Zinc Oxide (ZnO) nanocomposite silicone rubber by mechanical method. The dielectric properties of each sample were measured by dielectric spectroscopy. The experimental results showed that the dielectric constant of the silicone rubber composite increases with the increase of the content of nano-ZnO. Suresh *et al.* (2017), used the sol-gel technique in the chemical synthesis and characterizations based on electrical studies of pure and Ag-doped zinc oxide (ZnO) nanoparticles. The dielectric studies proved that both the dielectric constant reduces as the frequency increases.

From the review above, so many methods of preparation were chosen but only Banerjee *et al.* (2012), used solid state which is very cheap and environmentally friendly. The final product remained in solid form and structurally pure with the desired properties. Instead of Barium strontium titanate as in the case of Banerjee *et al.* (2012), ZnO is doped with NiO in this research and among other advantages of ZnO are significant physical and chemical stabilities, high catalytic

activity, effective antibacterial and bactericide function, and intensive ultraviolet and infrared absorption.

## II. EXPERIMENT

### A. Sample Preparation

In this work, the solid state method is used in the preparation of a doped zinc oxide. The materials used for the synthesis are 100 g of Zinc oxide (99.7% purity) and 50 g of Nickel oxide (99.7% purity) all in powdered form. The Zinc oxide was obtained at Halishuaib Chemicals while the Nickel oxide was obtained at CEMAN Chemicals Ventures. During the preparation of the composites, 5 g of Nickel oxide was mixed with 25 g of Zinc oxide using pestle and mortar, the combination was grinded continuously using mortar and pestle for about 60 minutes for perfect homogeneity. The mixed sample was taken to furnace and was heated to about 1000°C for appreciable reaction to take place. The same procedure was applied for 7.5 g of Nickel oxide and 22.5 g of Zinc oxide, 10 g of Nickel oxide and 20 g of Zinc oxide, 12.5 g of Nickel oxide and 17.5 g of Zinc oxide, and 15 g of Nickel oxide and 15 g of Zinc oxide. Five different mixtures with different proportions of Zinc oxide and Nickel oxide were prepared. The summary of materials composition is

shown in Table 1. The prepared composites are then ready for characterization.

TABLE 1: COMPOSITION OF SAMPLES

Sample	NiO		ZnO		Total	
	(g)	%	(g)	%	(g)	%
A	5.0	16.7	25.0	83.3	30.0	100
B	7.5	25.0	22.5	75.0	30.0	100
C	10.0	33.3	20.0	66.7	30.0	100
D	12.5	41.7	17.5	58.3	30.0	100
E	15.0	50	15.0	50	30.0	100

### B. Characterization

Characterization of the samples for dielectric constant were carried out using open ended coaxial probe HP85071C within a microwave frequency range of 8.2 to 12.2 GHz. The bonding and IR absorption of the samples were studied using FTIR within the range of 750 to 4000  $\text{cm}^{-1}$  wavenumber.

## III.

## IV. RESULTS AND DISCUSSION

### A. Dielectric Constant

After measuring all the samples under study, the raw data obtained was then used to plot the dielectric constant. Shown in Fig. 3.1 is the comparison of the dielectric constant obtained for all the samples.

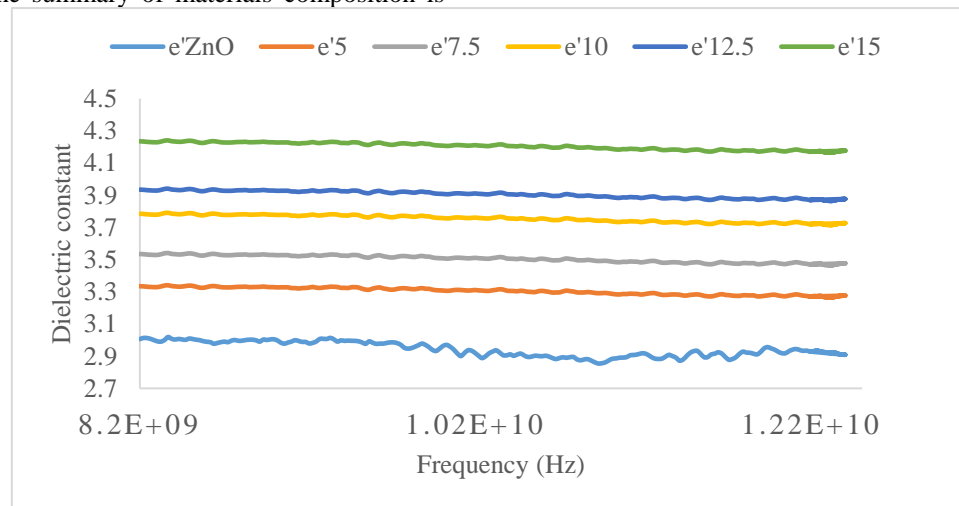


Fig. 3.1: Comparison of dielectric constant for pure ZnO and doped ZnO composites

The graph shown in Fig. 3.1 depicts a sequential increase in dielectric constant as pure ZnO is doped incrementally with the filler (NiO). The results shows a total increase in dielectric of 1.22 representing 40.0 %. Further observation shows that dielectric constant decreased as frequency increases for all samples. This result is in agreement with (Ahmad *et al.*, 2017), who observed in his research an increase in the dielectric properties of polymer as it is doped with nikel-ferrite. The same thing was also confirm by Sylvestre *et al.* (2007), who reported that the dielectric constant of Nickel/hydrogenated amorphous carbon dropped from 300 to 200 as the frequency increased from 1 Hz to 10 kHz.

At 8.2 GHz, it was observed that the pure ZnO have a dielectric constant of 3.01, 5 g doped ZnO have 3.33, 7.5 g doped ZnO have 3.53, 10 g doped ZnO have 3.78, 12.5 g doped ZnO have 3.93 and 15 g doped ZnO have 4.23. Similarly, at 10.2 GHz, it was observed that the pure ZnO have a dielectric constant of 2.91, 5 g doped ZnO have 3.31,

7.5 g doped ZnO have 3.51, 10 g doped ZnO have 3.76, 12.5 g doped ZnO have 3.91 and 15 g doped ZnO have 4.21. This shows an increase in dielectric constant as NiO is added (Sylvestre *et al.*, 2007) to the ZnO. It also revealed a decrease in dielectric constant as the frequency increases. The decrease in dielectric constant with frequency indicates dielectric dispersion is at low frequency region which is due to Maxwell-Wagner type of interfacial polarization in agreement with Koop's phenomenological theory (Yakubu *et al.*, 2015). The dielectric constant decreases rapidly at low frequencies and becomes quite slow at high frequencies. The higher value of dielectric constant at lower frequencies is explained on the basis of space charge polarization (Aldar *et al.*, 2014; Muhammad *et al.*, 2012). However, Sourav *et al.* (2014) reported that high value of dielectric constant at low frequency and almost constant value at higher frequency is due hopping contribution. Yakubu *et al.* (2015), reported that high value of dielectric constant at lower frequencies is

attributed to the interfacial ionic polarizations due to localized ion motion within the sample.

#### B. FTIR

Fig. 3.2 and 3.3 are the results obtained from FTIR spectroscopy for the 41.7 %, and 50.0 % doped ZnO

composites, respectively. They were acquired in the range of 750 to 4000  $\text{cm}^{-1}$  wavenumber, with a maximum transmittance of 100 %.

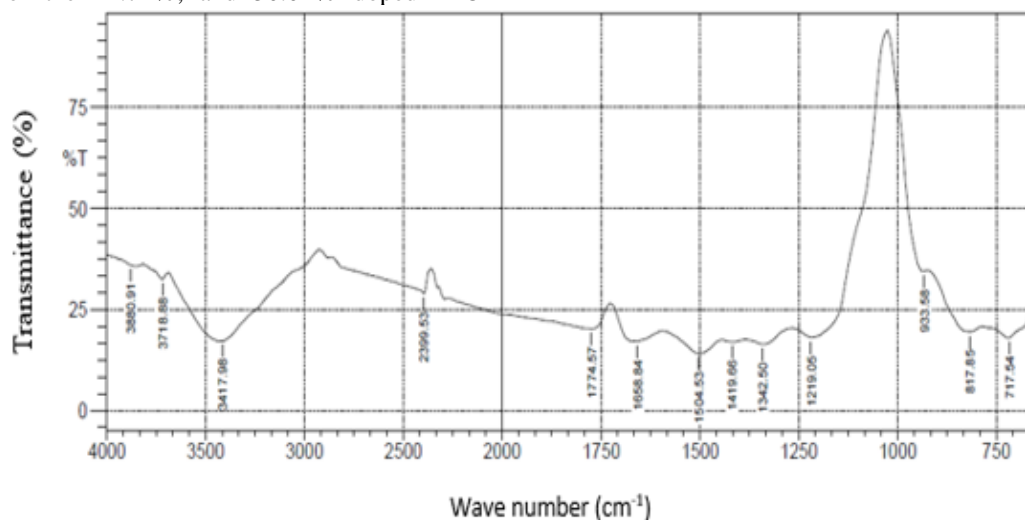


Fig. 3.2: FTIR Spectra for 41.7 % Doped ZnO

The FTIR spectrum in Fig. 3.2 shows the absorption region of 717.54  $\text{cm}^{-1}$ , 817.85  $\text{cm}^{-1}$ , 1219.05  $\text{cm}^{-1}$ , 1342.50  $\text{cm}^{-1}$ , 1419.00  $\text{cm}^{-1}$ , 1504.53  $\text{cm}^{-1}$ , 1658.84  $\text{cm}^{-1}$ , 1774.57  $\text{cm}^{-1}$  and 3417.90  $\text{cm}^{-1}$ . The highest absorbance observed is 86 % at 1504.53  $\text{cm}^{-1}$  corresponding to 14% transmittance. The absorption band observed at 717.54  $\text{cm}^{-1}$  corresponds to strong C-Cl stretching and the one at 1219.05  $\text{cm}^{-1}$  corresponds to C-N stretching vibrations. The band

appearing at 1342.50  $\text{cm}^{-1}$  attributed to C-H bending vibration. The band at 1419.00  $\text{cm}^{-1}$  corresponds to asymmetric C-H bending. The band at 1658.84  $\text{cm}^{-1}$  corresponds to the symmetrical of C=O. The band at 1774.57  $\text{cm}^{-1}$  attributes the strong C=O stretching and the one at 3417.90  $\text{cm}^{-1}$  corresponds to strong O-H stretching (Karthikeyan *et al.*, 2017).

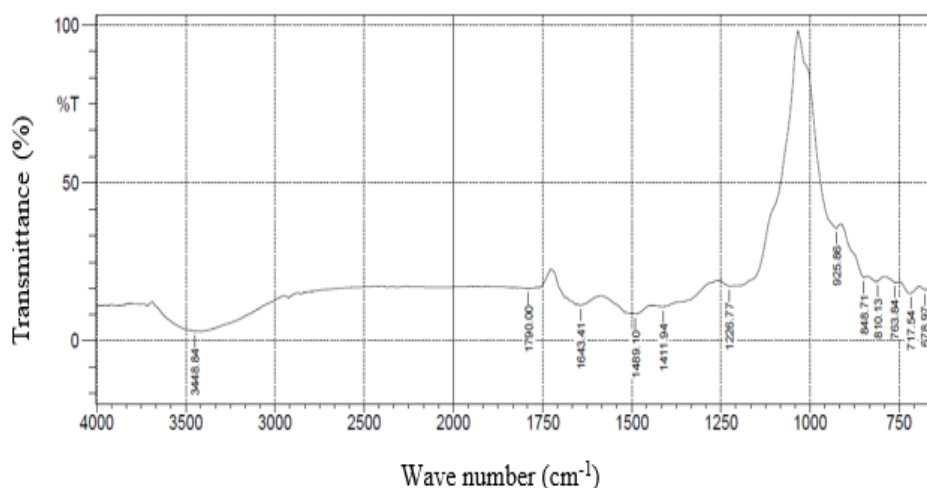


Fig. 3.3: FTIR spectra for 50.0 % Doped ZnO

Fig. 3.3 shows the absorption region of 678.97  $\text{cm}^{-1}$ , 717.54  $\text{cm}^{-1}$ , 763.84  $\text{cm}^{-1}$ , 810.13  $\text{cm}^{-1}$ , 846.71  $\text{cm}^{-1}$ , 1226.77  $\text{cm}^{-1}$ , 1411.94  $\text{cm}^{-1}$ , 1489.10  $\text{cm}^{-1}$ , 1643.41  $\text{cm}^{-1}$ , 1790.00  $\text{cm}^{-1}$  and 3445.84  $\text{cm}^{-1}$ . The highest absorbance observed is 97.1 % at a peak 3445.84  $\text{cm}^{-1}$  which corresponds to 2.9 % transmittance. The absorption band observed at 678.97  $\text{cm}^{-1}$  corresponds to O=C=O bending. The absorption band observed at 717.54  $\text{cm}^{-1}$  and 763.84  $\text{cm}^{-1}$  corresponds to C-Cl stretching. The band at 810.13  $\text{cm}^{-1}$  and 846.71  $\text{cm}^{-1}$

attributes C-C skeletal vibrations. The absorption at 1226.77  $\text{cm}^{-1}$  corresponds to C-O stretching vibrations. The band at 1411.94  $\text{cm}^{-1}$  and 1489.10  $\text{cm}^{-1}$  corresponds to asymmetric C-H bending. The band at 1643.41  $\text{cm}^{-1}$  corresponds to the symmetrical of strong C-H. The band at 1790.00  $\text{cm}^{-1}$  attributes the C=O stretching and 3445.84  $\text{cm}^{-1}$  attributes O-H stretching (Ajai & Sangshetty, 2017). The 50 % doped composites showed the highest absorption which indicates increase in absorption as doping quantity increases.



### C. SEM

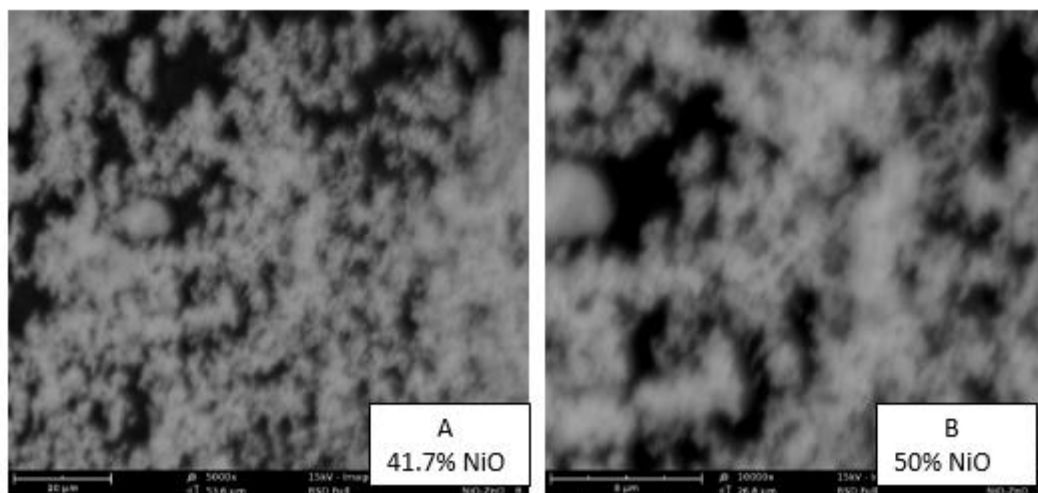


Fig. 3.4: SEM micrographs for doped composites

Fig. 3.4(A) shows a micrograph with whitish colour image agglomerated with dark dots of NiO (Karthikeyan *et al.*, 2017). Due to the density of the sample in Fig. 3.4(A), more light were able to pass through it which is evident in the FTIR analysis. Observation on Fig. 3.4(B) shows that the zinc oxide and filler are sparsely dispersed through the matrix of the composite materials, and visible traces of white and black coloring are seen indicating the equal percentage of mixture in the sample (Yakubu *et al.*, 2015).

### V. CONCLUSION

The synthesis of a doped ZnO composites using simple solid stated method was successfully carried out in this research. Based on the objectives of this research, investigations were carried out for the dielectric constants using open ended coaxial probe (OECF), FTIR for bonding and IR absorption and SEM for morphology. From the result, it was found that as NiO content is increased, both dielectric constant and IR absorption increases and the more the particles agglomerate. It is therefore concluded that NiO can be used as a filler for improving dielectric constant with ZnO as a matrix. The composite (NiO/ZnO) can serve as a good agent for constructing capacitors and other dielectric based materials that can be used in constructing electronic and telecommunication gadgets.

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