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RESEARCH ARTICLE

Preparation and Characterization of $SrFe_{12}O_{19}/ZrO_2$Nanocomposite by using Sol-Gel method.

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Abstract

Strontium hexaferrite and Zirconium oxide ($SrFe_{12}O_{19}$ - ZrO_2) nanocomposite were prepared by sol-gel method in different weight percentage. In this method Strontium nitrate, iron nitrate and zirconium oxy chloride were used as a raw materials and glucose acted as a fuel. Fourier transforms infrared spectroscopy (FTIR). The wave number 453-511cm-^{1} indicated the presence of the ferrites. Thermogravimetry analysis (TGA/DTA) curve above 850^{0}C confirming the formation of the stable Strontium hexaferrite. The $SrFe_{12}O_{19}$ - ZrO_2phase structure, functional groups were analyzed by X-ray diffraction (XRD) the formation of pure crystallized nanocomposite occurred when the precursors were calcined at 800°C for 3 hours. The morphology and quantitative analysis of the prepared particles were studied by using SEM and EDX spectrum. M-type strontium hexaferrite magnetic resonance frequency range is 11.51 GHz.
Strontium hexaferriteconfirms that the material can be considered for high frequency applications.

Introduction:-

Strontium hexaferrite is considered to be a scientifically favorable material for technological operations in the field of Nanoscience devices. Its high magnetic anisotropy, electrical resistivity, chemical stability and corrosion resistance makes it more suitable to be used as ferrites5. [1] .M-type hexaferrite, due to its perfect chemical stability, good thermal durability, corrosion resistivity, unique electrical and magnetic properties, has extensive applications in permanent magnet, high density magnetic recording devices, telecommunication, magneto optical and microwave devices [2]. Strontium hexaferrite ($SrFe_{12}O_{19}$) has interesting chemical, physical and magnetic properties in addition to high performance-to-cost ratio [3]. In order to improve the function of ZrO_2 some magnetic materials must be combined with ZrO_2 combining with the advantage of SrM and ZrO_2 in the promising novel system can provide a new functional with synergetic or complementary with behavior between each constituent.[4]

2. Experimentalprocedure:-

($SrFe_{12}O_{19}$-ZrO_2) nanocomposites are prepared by using sol-gel method in different weight percentage. Appropriate amount of reactive oxidants such as $Sr(NO_3)_2$, $Fe(NO_3)_3.9H_2O$, $ZrOCl_2$ get dissolved into an unionized distilled water , then add glucose which is a fuel and it gives requisite energy to initiate exothermic reaction and add ethylene glycol which is act as a reducing agent . The solution mixture is stirred continuously for 2 hours at 80^{0}C. As water evaporated, the solution become viscous and finally formed a very viscous brown dried gel, it again dried at110^0 C

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for 24hours to get a precursor powder which is used to take FTIR. Then dried powder is again calcined at 800^0C for 3hours in a furnace.

Results AndDiscussion:-

3.1 Thermogravimetry Analysis (Tga/Dta):-

The thermo gram of the precursor of strontium hexaferrite derived by mixing of strontium nitrate, ferric nitrate and glucose as shown in **Figure (1).**TGA shows the initial weight loss from 25°C to 165 °C due to the loss of absorbed water. The subsequent loss up to 316°C is associated mainly to the decomposition of the glucose. In order to verify this, separate TGA was undertaken for glucose. It shows the major weight loss between 389°C and 565°C thus supporting our assignment. Therefore, glucose provides self heat to promote the reaction and to reduce the crystallization temperature of the hexaferrite. The stage of decomposition between 565°C and 704°C is due to decomposition of nitrates and starting formation of hexaferrite. There is no considerable weight loss above 850°C, confirming the formation of the stable Strontium hexaferrite this analysis, therefore illustrates the optimum calcinations temperature for Strontium hexaferrite is around 850°C.

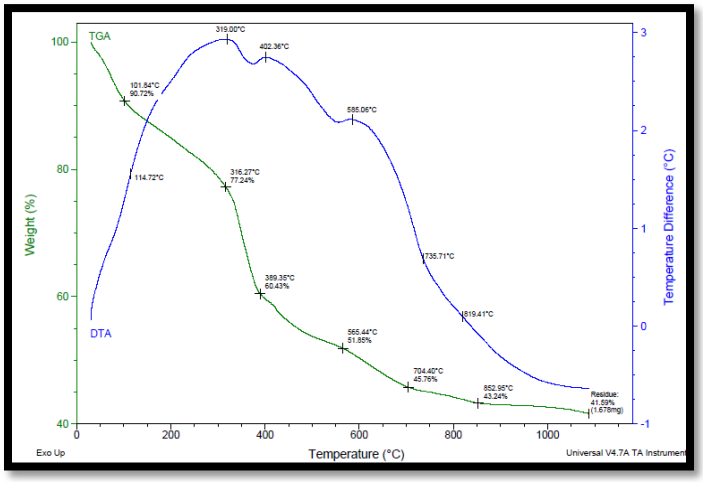


Fig .1:-TG-DTA Curve for precursor at 800^0C

3.2 Vibrational studies:-

Fourier Tran form infrared spectroscopy:-

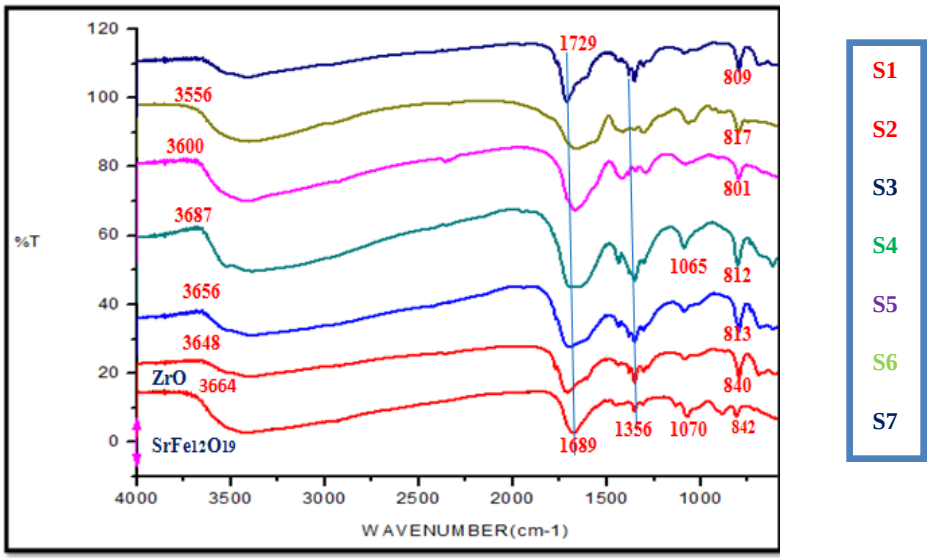


Fig.2:-FTIR spectrum of ($SrFe_{12}O_{19}$ – ZrO_2) nanocomposite

The FTIRSpectrums of the ($SrFe_{12}O_{19}$ – ZrO_2) samples are shown in fig (2). The absorption peak at very large band around 3664 cm^{-1} is attributed to the stretching of O-H stretch, the sharp absorption peak corresponding to C=O stretch was absorped1729 to 1689 cm^{-1}. The small absorption peaks at C-O stretch bend 1356 to 1065cm^{-1}. The strong absorption peak between 551cm^{-1}-842 cm^{-1} belongs to the metal oxide stretching vibration of ($SrFe_{12}O_{19}$ – ZrO_2).[5]. The wave number 453-511cm^{-1} indicated the presence of the ferrites. .FTIR (Fourier Transform Infrared Spectroscopy) analysis was used to determine the functional groups of strontium hexa ferrite and zirconium oxide nanocomposites.

Table 1:-FTIR spectrum of ($SrFe_{12}O_{19}$ – ZrO_2) nanocomposites

SAMPLE	WEIGHT RATIOS (%)	WEIGHTPERCENTAGE(CM^{-1})	VIBRATIONAL MODE(CM^{-1})
S1($SrFe_{12}O_{19}$)	**100:100**	**3664**	**O-H Stretch**
S2(ZrO_2)	**100:100**	**1729-1689**	**C-O Stretch**
S3($SrFe_{12}O_{19}$+ZrO_2)	**90:10**	**1356**	**C-O Stretch**
S4($SrFe_{12}O_{19}$+ZrO_2)	**70:30**	**1065**	**C-Stretch**
S5($SrFe_{12}O_{19}$+ZrO_2)	**50:50**	**842**	**Metal oxide**
S6($SrFe_{12}O_{19}$+ZrO_2)	**30:70**	**551**	**Metal oxide**
S7($SrFe_{12}O_{19}$+ZrO_2)	**10:90**	**453-511**	**Ferrites**

3.3 Structural studies:-

X-ray Diffractiion:-

It shows the XRD patterns of the$SrFe_{12}O_{19}$ – ZrO_2 nanocomposites obtained by sol-gel method in different weight ratios. The major phase corresponds to (110), (111), (200), (220), (311) were found to be matched which confirmed the presence of hexagonal structure nanocomposites. In the XRD paths of $SrFe_{12}O_{19}$ – ZrO_2 nanocomposites diffraction peaks 30^0, 32^0, 35^0, 38^0, 52^0, 62^0can be assigned to hexagonal structure. The $SrFe_{12}O_{19}$(JCPDS NO: 841531) and ZrO_2 (JCPDS NO: 781807). Well defined sharp peaks in the XRD pattern of sample indicates the good crystalline quality and confirm the formation of nanocomposites the average particle sizes of different phases were determined from the line widths of diffraction peaks using Scherer equation.

$$D=K\,\lambda/\beta\cos\theta$$

D- Grain diameter.

B- Half intensity width of relevant diffraction.

λ- X-ray wavelength.

θ- Diffraction angle.

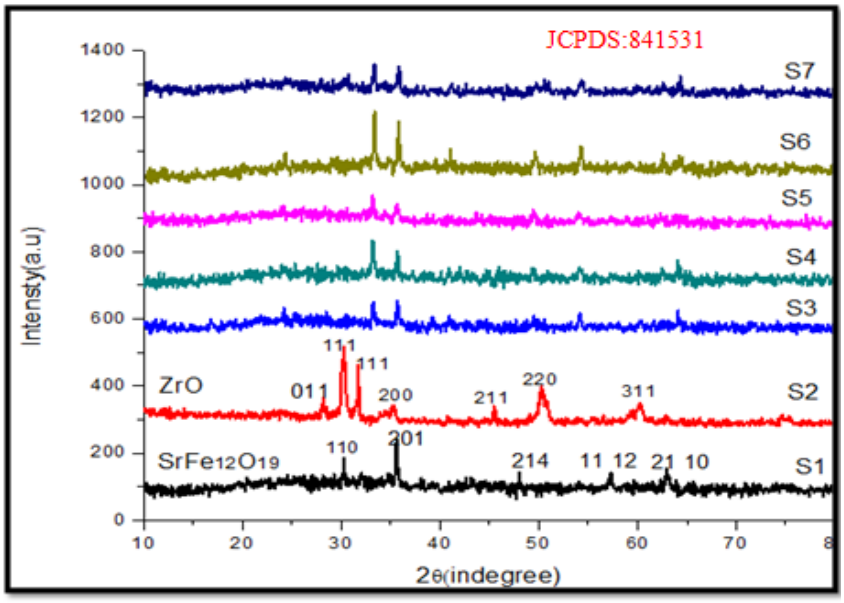


Fig.3:-XRD pattern of ($SrFe_{12}O_{19}$ – ZrO_2) nanocomposite at 800^0C

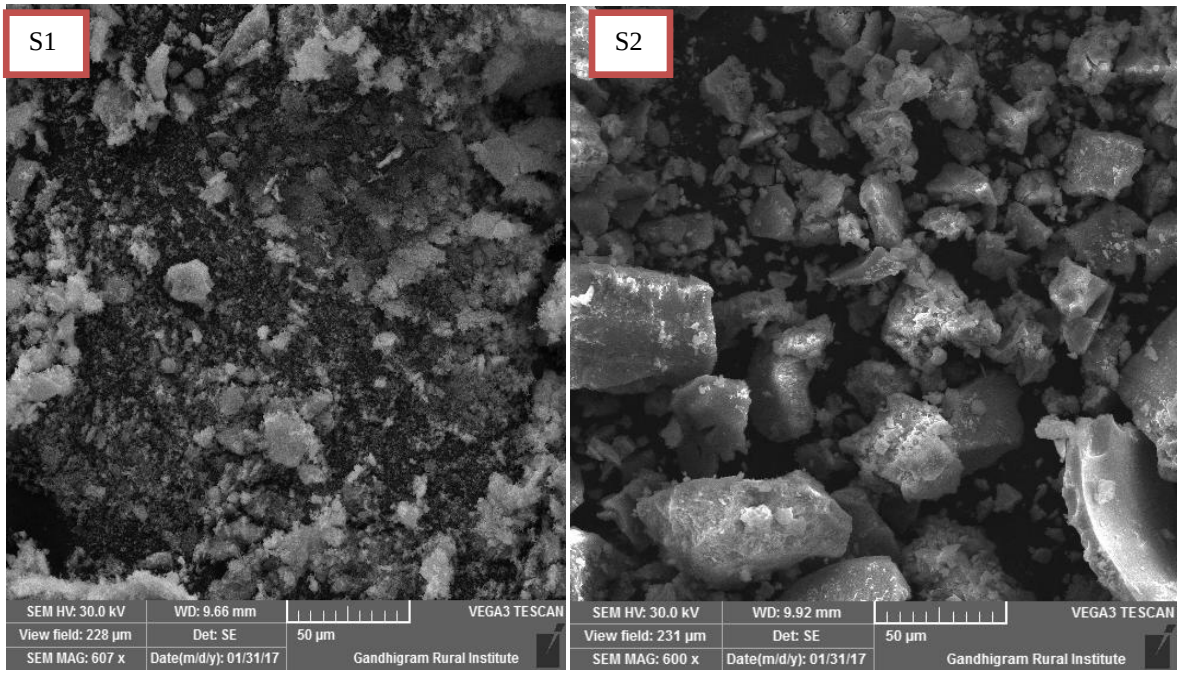
Table 2:-Particle size of various samples at 800^0C

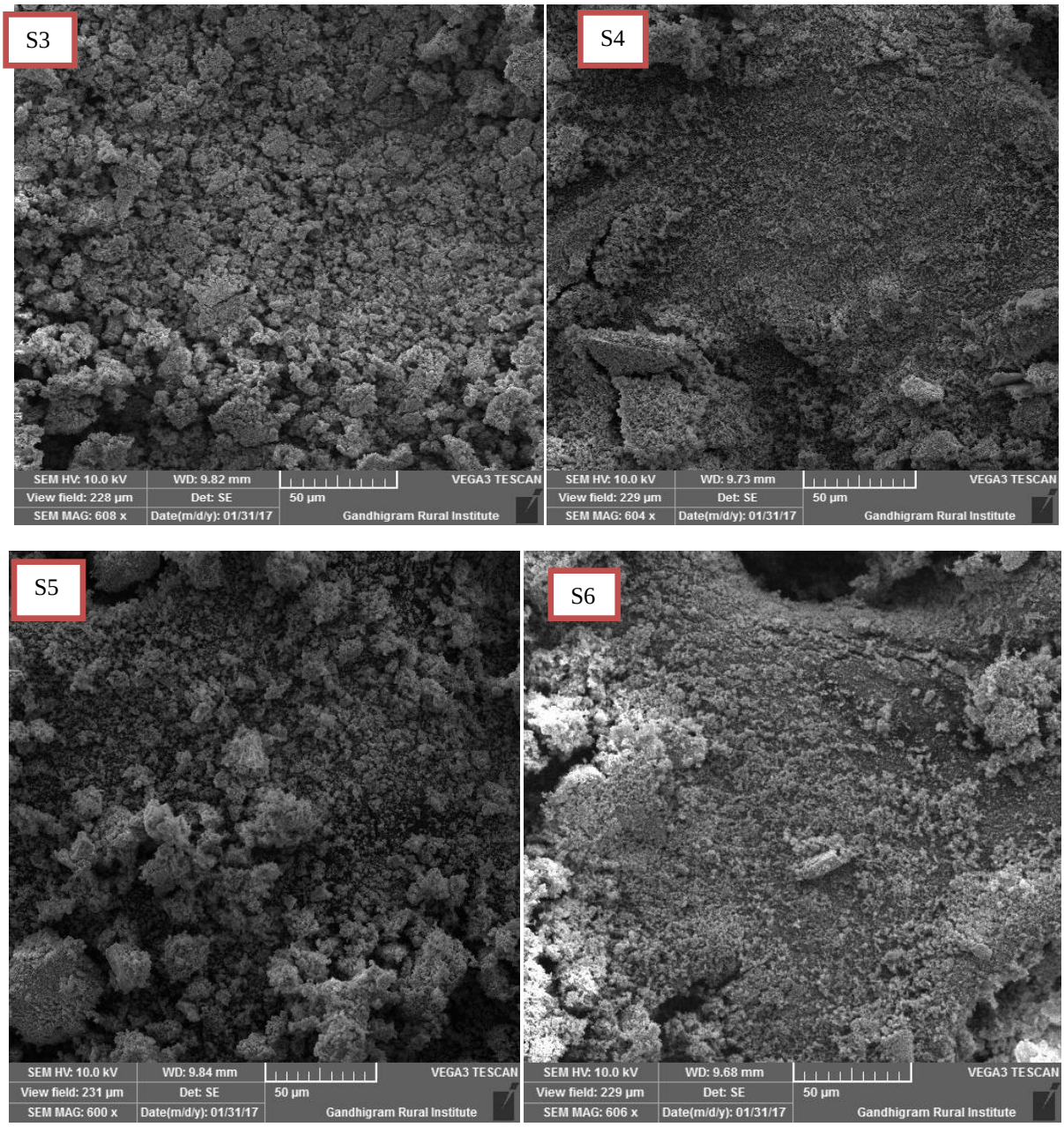
SAMPLE	WEIGHT RATIOS (%)	PARTICLE SIZE(nm)
S1($SrFe_{12}O_{19}$)	**100:100**	**26.39**
S2(ZrO_2)	**100:100**	**30.89**
S3($SrFe_{12}O_{19}$+ZrO_2)	**90:10**	**25.25**
S4 ($SrFe_{12}O_{19}$+ZrO_2)	**70:30**	**20.52**
S5 ($SrFe_{12}O_{19}$+ZrO_2)	**50:50**	**22.58**
S6 ($SrFe_{12}O_{19}$+ZrO_2)	**30:70**	**23.56**
S7 ($SrFe_{12}O_{19}$+ZrO_2)	**10:90**	**27.98**

3.4 Morphological Studies:-

Scanning Electron Microcope:-

Figure **(4)** the morphology of strontium hexaferrite with zirconium oxide nano composite used for different weight ratios of (100, 100, 90/10, 70/30, 50/50, 30/70, 10/90) powder calcined at 800^0 C for 3h Strontium hexa ferrites with zirconium oxide up to 30% weight ratios the particle sizes were decreased. Above 50 to 90% strontium hexa ferrite with zirconium oxide weight ratios the particle sizes were increase. The XRD and SEM results were matched. [6]




S3
SEM HV: 10.0 kV
WD: 9.82 mm
VEGA3 TESCAN
View field: 228 µm
Det: SE
50 µm
SEM MAG: 608 x
Date(m/d/y): 01/31/17
Gandhigram Rural Institute
S4
SEM HV: 10.0 kV
WD: 9.73 mm
VEGA3 TESCAN
View field: 229 µm
Det: SE
50 µm
SEM MAG: 604 x
Date(m/d/y): 01/31/17
Gandhigram Rural Institute
S5
SEM HV: 10.0 kV
WD: 9.84 mm
VEGA3 TESCAN
View field: 231 µm
Det: SE
50 µm
SEM MAG: 600 x
Date(m/d/y): 01/31/17
Gandhigram Rural Institute
S6
SEM HV: 10.0 kV
WD: 9.68 mm
VEGA3 TESCAN
View field: 229 µm
Det: SE
50 µm
SEM MAG: 606 x
Date(m/d/y): 01/31/17
Gandhigram Rural Institute

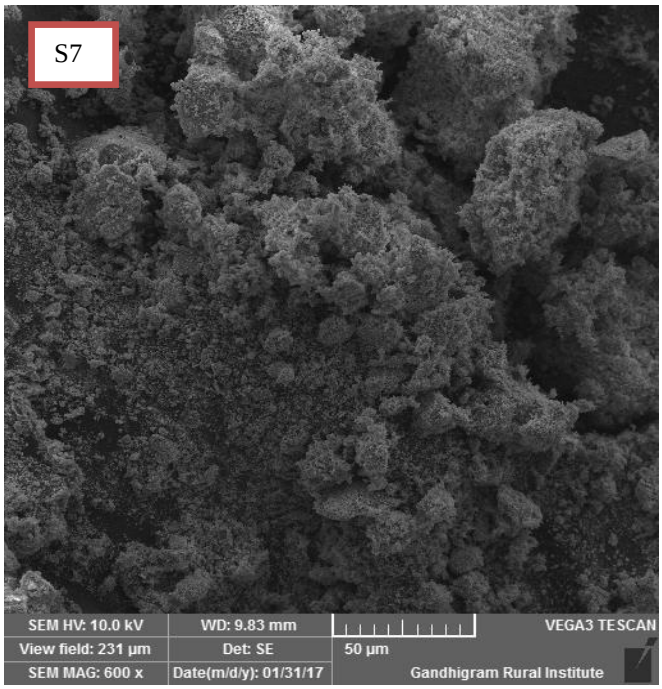


Fig .4:-Sem Image Of ($Srfe_{12}O_{19}$ –Zro_2) Nanocomposites At 800^0c

3.5 Edax (Energy Dispersive X-Ray Spectroscopy):-

The representative EDX spectra clearly show that presence of strontium, iron, zirconium, oxygen.

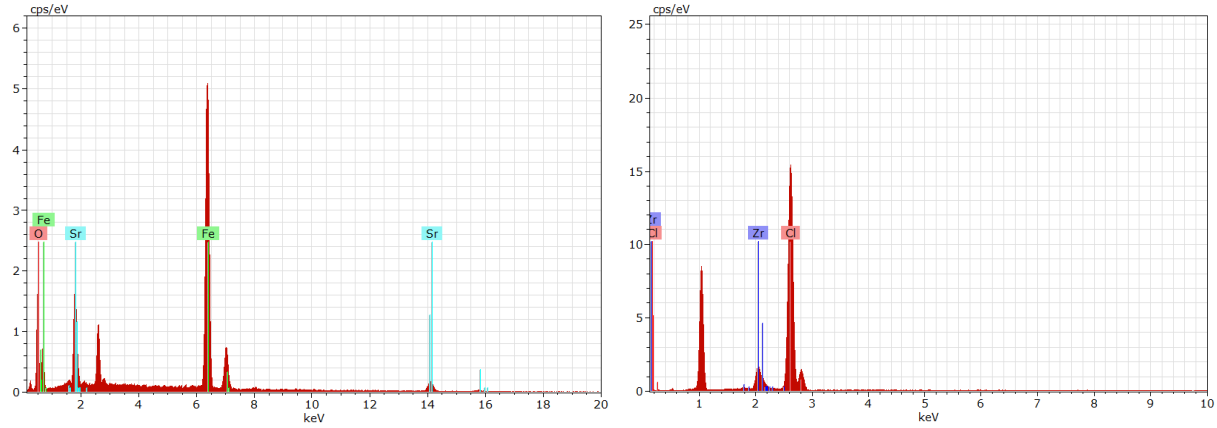


FIG .5:- (a) EDX image of $SrFe_{12}O_{19}$**Fig .5:- (b)** EDX image of ZrO_2

3.6 Dielectric Property:-

Table 3:-Strontium Hexa ferrites dielectric property

Sample Name	Frequency (GHz)	ε_r	Tanδ (10^{-3})
S1 (1100 ˚C/ 3 hrs)	11.51225	19.25	2.426
S3, S4, S5 (1100 ˚C/ 3 hrs)	didn't get any resonance due to non-sinterability		
S4, S6 (1200 ˚C/3 hrs)	didn't get any resonance due to non-sinterability		

The dielectric constant range **20 (ε_r).**
The dielectric loss range 0.002426 (Tanδ).
The frequencies range 11.51 (GHz).
The material shows low loss and high frequency material. [7]

Conclusion:-

Thermogravimetry analysis (TGA/DTA) curve absorber weight loss 850^0C confirming the formation of the stable Strontium hexaferrite. Fourier transform infrared spectroscopy (FTIR) spectra of ($SrFe_{12}O_{19}$- ZrO_2) 453-511cm-^{1} indicated the presence of the ferrites. X-ray diffraction (XRD) average crystallite sizes of the particles were 20-31 nm. In accordance with the SEM graphs average particle size increase and decrease as the amount of weight ratios increase. The EDX indicated the presence of (Sr, Fe, Zr, and O). M-type strontium hexaferrite magnetic resonance frequency range is 11.51 GHz. Strontium hexaferriteconfirms that the material can be considered for high frequency applications.

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