RESEARCH ARTICLE OPEN ACCESS

Impact of POT (poly-ortho toluidine) coating on synthesis and characteristics of Fe_3O_4 (Iron oxide nanoparticles)

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Abstract:

The structural, morphological, the measurement of the energy of characteristic X-ray emissions of excited specimens and microwave absorbing properties were examined by using XRD, SEM, EDS and testing source network Analyser, respectively. The shielding results indicated that Fe_3O_4 were coated with polymer chains of POT and interfaces between them. The results were created at the 0.19mm of thickness and acquired a minimum reflection (RL) of -16dB at 1.99 GHz with the absorption loss of -2.39 dB. Moreover, the maximum achieved value for the maximum and minimum absorption loss are perceived to be 6.1dB and 3.0dB with the collective effect of shielding 9 dB at 17GHz. It is determined that shielding response of composite, enhanced with the frequency increase and vice versa. These consequences demonstrate the material retain modest shielding performance and can be authorized as EMI shielding material owing to its favourable microwave frequency at merely 0.199 mm of thickness.

Keywords: **Electromagnetic shielding interference (EMI), Polymer composite, Iron oxide nanoparticles (IONPs), Poly-ortho toluidine (POT)**

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I. INTRODUCTION

The innovation in the field of electronics swayed attention towards the impoverishment of the devices that the devices are getting smarter and made smaller day by day [1]. The significant usage of up-to-date electronic devices also stimulated definite kinds of radiations like electronic blare, electromagnetic (EM) waves, fallouts and radio -frequency radiations as the consequence [2]. Electromagnetic waves are nominated into the spectrum allowing to their energy and frequency. The consumption of the radiations reliant on the nature and category of the device [1]. These unwanted radiations cannot be unrestrained because they unwaveringly affect with the restrained apparatuses or the neighboring devices to diminish the competence of the device, this kind of interference is labelled as the electromagnetic interference (EMI) [2, 3]. In moderate words EMI is defined as the kind of electronic noise that can interpose, lower and deter the normal operation of the components or the devices [4]. Metals the most frequently and preferably used as EMI shielding material because of in arrears to remarkable EMI shielding efficiency (SE) [5]. Conservatively used metallic material as shields were bulk sheets, interlocks, plating coatings, powders, fibers doped with polymer composites and coatings indicate too much weight consequences for the applications like space-craft and satellite system. Number of exertions are being done in this concern to lesser the weight of EMI shields by suggesting coatings of ceramic micro-balloons, carbon fibers and conductive fillers with metals. Still, there is further problem of corrosion between the linkages which ominously descents the performance of SE that prevent the procedure of metals as EMI shields.

The importance of the EMI shielding material is directly dependent over the reliability of electronic arrangement and wireless technology from the EM contamination [1].This expansion of the EM pollution depreciate the environment and electronic devices if no shielding is provided to block these harmful radiations [1, 6]. Subsequently, there is urgent requirement of such materials that are operative EMI shields [1]. The design of EMI shielding material is for imposing to diminish the EM pollution. The material must cost effective and light weight to come across up all the contemporary requirements and also shield the constituents from being hindered from the stray radiations [7]. For that reason, EMI shielding is the finest technique to shield the environment, electronic devices or components and man's health from the harmful effects of EM waves in the martial and civil uses [8-10]. EMI shielding is the foremost concerned subject of the current era due to progression in technology and necessity of electronic devices that emit the radiations foundations [11].Likewise, the complication in electronic systems such as the high packaging density arise due to the frequent reaction and diminishment of the devices causes with the emission of worst kind of radiations which is improving the EM pollution professionally [12-14]. EMI shielding is the phenomenon that meet the development of emission, absorption, reflection and compound internal-reflection of radiations by the substantial. The material, that include of mutually electric and magnetic components, act as a shield to absorb all the radiations from the penetration of electromagnetic radiations into the electronic devices [15]. Metals the most frequently and preferably used as EMI shielding material because of their remarkable EMI shielding success (SE) [5]. Essentially conducting polymers (ICPs) gained admiration in the current time due to their outstanding electrical belongings that could be tunable to the filler which are being used for the development of the shielding material. Their properties that make them attractive are light-weight, physical flexibility, corrosion protection, ease of modifying and tunable shielding response [16]. Due to the essential limitations like electrical conductivity and dielectric properties, ICPs can be used as conducting fillers for many insulating matrices like conductive, dielectric and magnetic [3]. Their special behaviors such as amending electrical conductivity, permittivity, permeability makes them as the finest innovative applicants for abundant techno -commercial applications. Their applications are also massive range in the progressive fields like astronomical, defense, triangulation control and as an absorbing material in radar technology [3, 17-19]. A deep explorative study in the already surviving EMI shielding material releases that there is no single -phase material yet equipped which indicated aspirational enactment under different conditions and applications. Their response to change in properties like thickness, absorption, volume and wide frequency band spectrum. That's why scientific communal made numerous kinds of struggles to prepare amalgamations of ICPs with conducting and magnetic material to prepare blends, mixtures and compounds [3, 11, 20]. Amongst the earlier stated combination, composite gained absorbing inspection owing to captivating properties and wealth of prevalent claim. These composites were equipped with the help of organic and inanimate filler laden with ICPs matrix or the ICPs may loaded with the insulating matrix to acquire the desired performance level [3, 20]. At modern times, revelation of various nanomaterials (NMs) and the approaches to accustom their electrical and electromagnetic properties may able to find the finest material for shielding and further applications. In addition to this nanocomposite occupy enormous scientific attention due to having the discriminant set of properties together with the favourable candidate for numerous applications [3].The suitable way to overawed the problems of metallic & polymeric shielding is to make the polymer conducting [21]. For this resolution, conducting & magnetic fillers like carbon fibres, carbon nanotubes(CNTS), carbon black (CB), graphene nanoparticles, graphene oxide, fly ash [22-27], ferrites are preferred for EMI shielding application because of their high electrical conductivity, lightweight & flexibility [28, 29]..Till now certain experiments are necessary to be talked such as thickness, Flexibility, magnetic & dielectric properties, filler loadings at lowest concentration & absorption over wide band simultaneously [26, 30-32] .As a result, there is necessity of a composite consuming all above-mentioned properties completely for EMI shielding. For enlightening the absorption of radiations concluded binary dielectric, magnetic individualities, preserving flexibility, lightness, thickness & concentration of fillers at the similar time.

Purpose of present study is to fabricate the Fe_3O_4-Poly (o-toluidine) for enhanced EMI shielding. We are proposing that the synthesized nanocomposite is lightweight, flexible, with wide range of absorption frequency & thus a better choice for shielding application.

I. Materials and Method

A. Synthesis of IONPs

Improved version of co-precipitation method is used for IONPs. That was experimentally confirmed by the results in terms of suspension and morphology of NPs. Firstly, we prepared the solution of $FeCl_2.4H_2O$ (0.5M) and $FeCl_3.6H_2O$ (0.8M) with a weighted amount of 20mL with DI water. The pre-synthesized solution was further heated at 50°C for complete disintegration of the reactants with continuously stirring at 600rpm for about 25min. This above solution contained the iron (II) chloride: iron (III) chloride in molar ratio of about 1: 1.7. Separately they prepared the 20mL base solution of NH_3OH (1.6M) which as poured drop wise into the already prepared solution with stirring maintained at 800rpm and 50°C for about 25min. The reaction was confirmed with the formation of the black precipitate of IONPs that were remained suspended over the base solution with PH at 11. Finally, the black precipitates were collected via the permanent magnet and then washed four times to remove all the filtrates and to maintain the PH at 7.2. Then the particles were dried in an oven at 80˚C.

B. Formation of IONPs/POT polymer nanocomposite

Then 3ml o-toluidine (OT) monomer is mixed with 10ml Deionized water (DI) and sulphuric acid (H_2SO_4) were added into pre-synthesized solution of IONP's. And put on stirring for about 30 minutes. Moreover, 1M Ammonium per sulphate (APS) solution with (0.5 mol/L) were added drop wise for initiating of the reaction during the 1st hour of stirring. For reaction to proceed it is constantly stirred for 18h to complete polymerization. This composite is further treated with DI and filtered. The filtered composite was given washing 2 times and dried at 40°C temperature for 2 days. Then the composite was crushed into powdered form and subjected into desired shape and size for further characterization.

II. Results and Discussion

A. Structural Properties Analysis

The crystallinity and structure of as prepared *Fe_3O_4-POT* nanocomposite were examined by using X- ray diffraction. As shown in Figure, the XRD pattern obtained for the *Fe_3O_4-POT* nanocomposite. It was found that the main characteristic diffraction peaks with 2θ values 30°,38°,53°, and 62° corresponding to (220), (311), (440), and (533) planes respectively for *Fe_3O_4* which well matched with (JCPDS #41-1487). The peaks obtained at $2\theta = 20° - 40°$ showed the presence of the POT. The peaks at 20.2°, 35.3°, and 50.4° are three extensive and in some way noticeable and other are the frail that are covenant. Besides, the changes in Bragg's angle causes the shifting of peaks which is showing the transformed crystallographic structures. Also, the further small peaks indicate the impurities go through this nanocomposite during procession as at 70-75° and 40-45° etc.

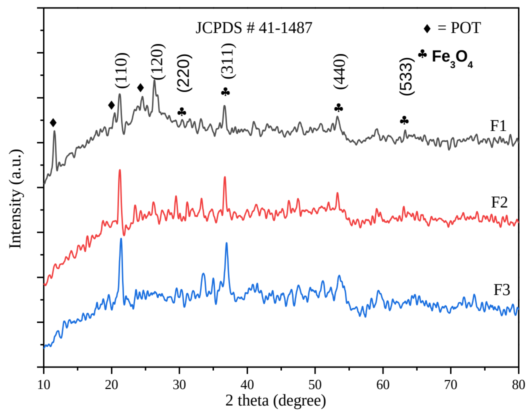

Fig. 1 Comparison of XRD patterns of samples F_1, F_2 and F_3 against angle and intensity

The Fig. 1 shows peaks of samples F1, F2 and F3 which illustrate the allover effect of POT over the iron oxide nanoparticles. This combination of overlapping of peaks showing the effect of POT with IONPs. The changes in peaks from their standard position showing the presence of POT and IONPs. These results are in approval for the efficacious grounding of the desire nanocomposite.

B. Energy dispersive spectroscopy (EDS)

Energy-dispersive X-ray spectroscopy is based on the measurement of the energy of characteristic X-ray emissions of excited specimens under study. Here we examine our nanocomposite, as in sample 1, we can see the relevant peaks of elements used in the synthesis and coated polymer. As presence of iron Fe, Cl observed in graph and peak of S is indicating the presence of POT.

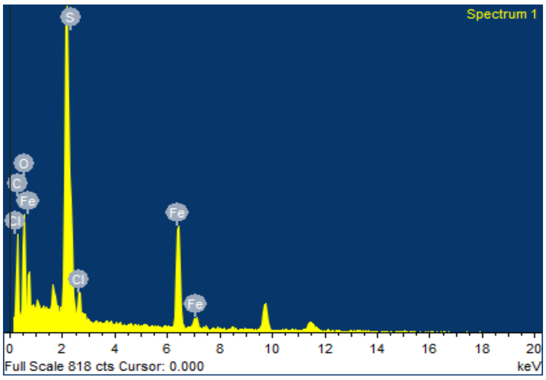

Fig. 2 EDS graph of sample 1

Similarly, in sample 2 we can see also see the peaks of Fe, Cl and afore-mentioned S is representing POT.

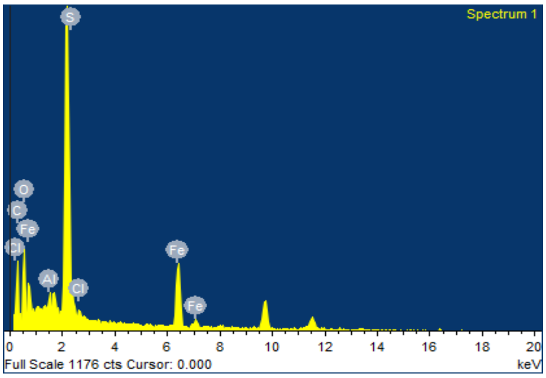

Fig. 3 EDS graph of sample 2

The below image is of sample 3, in which we also can see the relevant peaks of corresponding elements, as Fe, Cl and S.

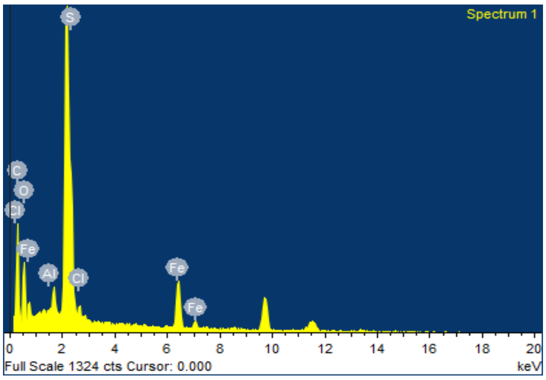

Fig. 4 EDS graph of sample 3

C. Scanning electron microscopy (SEM)

The surface morphology and accumulation behaviour of the POT coated IONPs is examined through SEM. Following figures shows the photographs of POT coated IONPs.

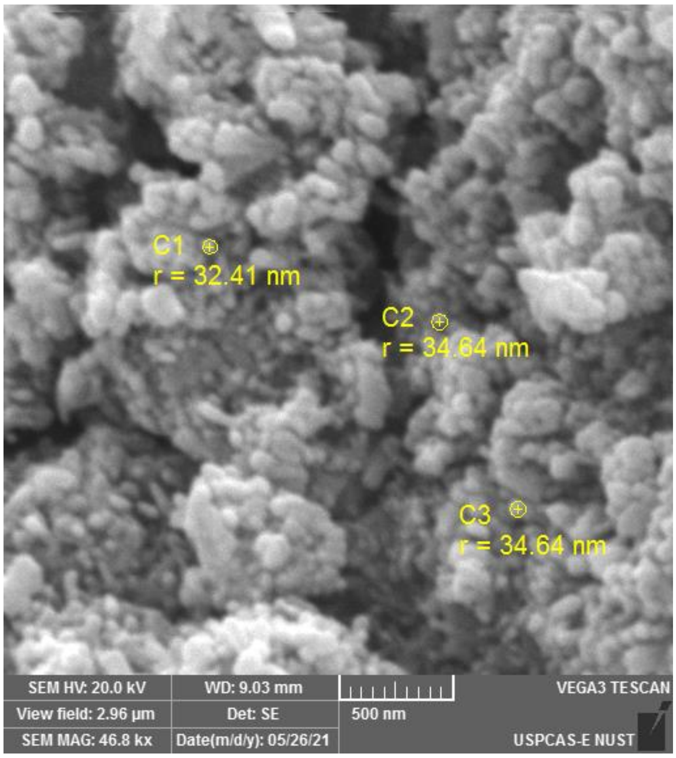

Fig. 5 SEM analysis at 500nm

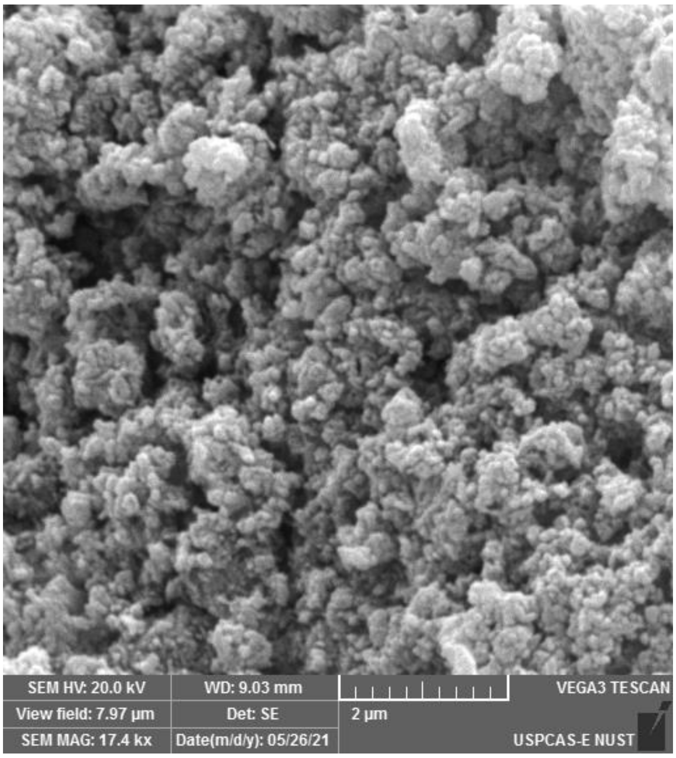

Fig. 6 SEM analysis at 2µm

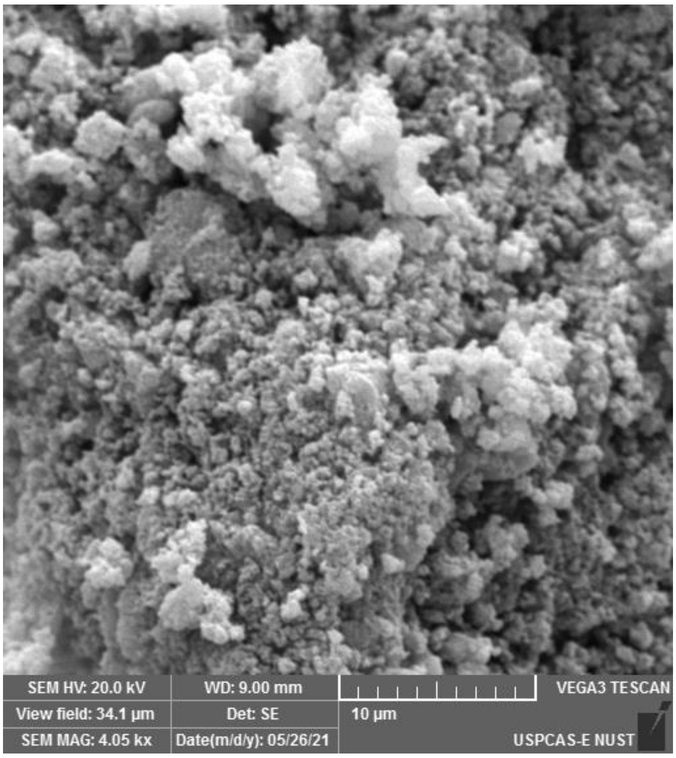

Fig. 7 SEM analysis at 10µm

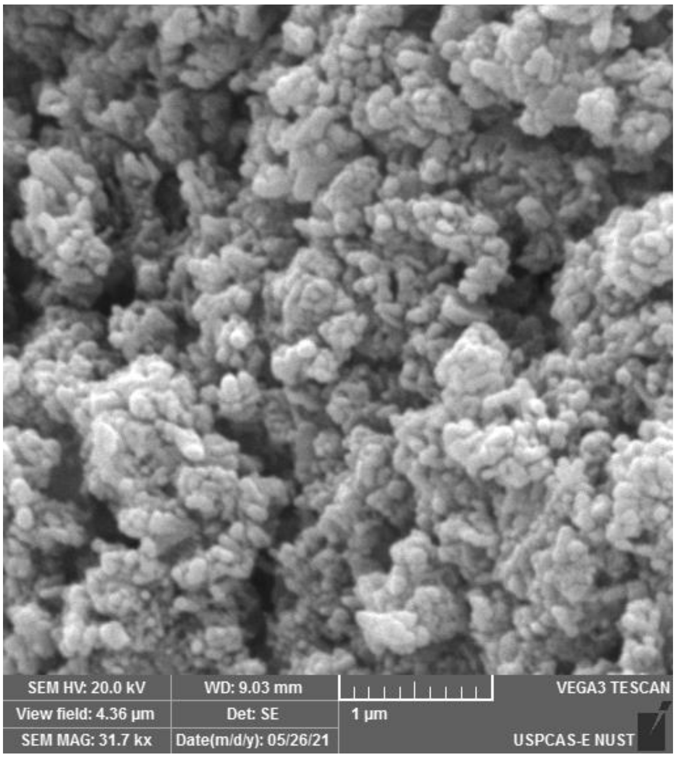

Fig. 8 SEM analysis at 1µm

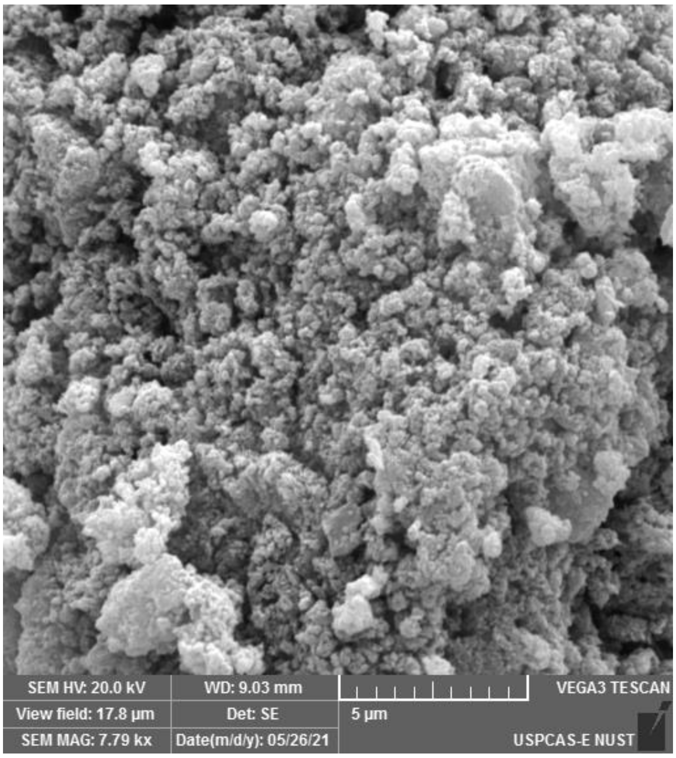

Fig. 9 SEM analysis at 5µm

These photographs of SEM are at 500nm, 2µm, 10µm, 1µm and 5µm. The aggregation of nanoparticles is due to their high magnetic properties and the interaction among them. The high surface charge and dipolar interaction caused these particles to form large clusters. The high surface energy and dipolar attraction forces are reduced due to the coating of polymer. It is also notice that all the samples shown interwoven fibrous structures.

D. Electromagnetic Shielding Interference (EMI)

The aforesaid polymer composite then made-up over the 6×6 Aluminium sheet having thickness of 1.4mm upon which we have coated our polymer composite with nearly 0.15mm thickness. That were investigated for their EMI shielding over (2-18) GHz bands. Fig. 10 shows the reflection, absorption and total EMI shielding response of composite with variation of the frequency.

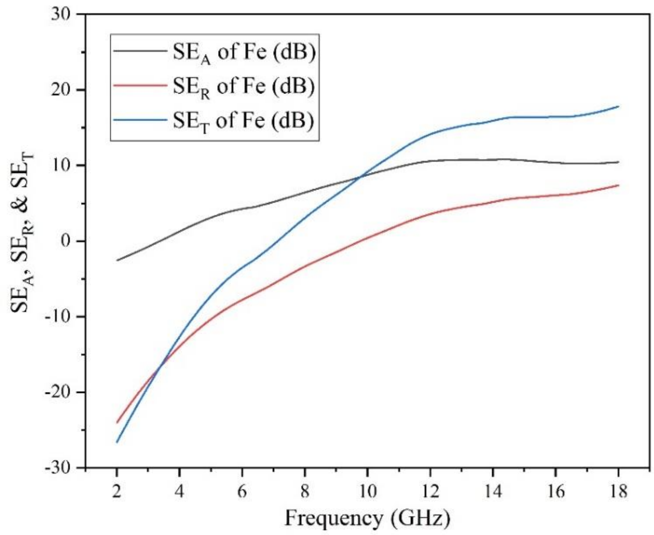

Fig. 10 Shielding performance IONPs with POT polymer composite with absorption, reflection and total (SE_A, SE_R and SE_T)

It can be seen that total Shielding Effectiveness is likewise shared by the separate components of SE_R and SE_A. The response of each component recognized with its nature and associated its GHz matching frequency. Consequently, penetration of IONPs and coated with (OT) monomer give rise to improved results. The shielding effectiveness accomplished at different frequencies resulted due to the addition of POT with the insulating -ferrite nanoparticles as they alone sedentary to produce shielding mechanism. According to previous works coating of OT over the Fe matrix has the improved effects over the shielding marvel. The response of each sample is illustrated in the graph mentioned in Fig. 10. the over-all tendency observed during the entire experiment were that the values of SE_R and SE_A somehow improved with the rise of frequency. The enhancement on the EMI values are consistent with the addition of POT content. Further, the coating of polymer enhanced the absorption mechanism. From the experimental data it is delineated that minimum reflection loss (RL) and absorption loss (AL) is observed to be (-16, -23) dB. furthermore, there would be a constant value of shielding but their individual component was changing with frequency. It is also noted that at lower frequencies SE_T dominated due to reflection loss and further rise in bands of frequency leads dominated phenomenon of absorption at minimum matching thickness of approximately of 0.199mm that were kept constant during the whole experiment. The result of the filling upon the composite shielding effectiveness is somehow similar as shown in the figures with a very little change but their individual components SE_A and SE_R are not alike as both absorption and reflection loss are related with the essential performance of magnetic and dielectric elements added in the composite. Even though it is very thought-provoking task to associate their EMI properties since every amalgamation has strange behaviour upon the incident radiation due to its wt.% content, polymer matrix type and many other uncontrolled conditions like preparation method, dried conditions, different dopant etc. This manufactured polymer composite (FP) consuming modest values.

III. CONCLUSION

In present research work, we successfully synthesized Fe_3O_4-Poly (o-toluidine) by in situ polymerization process. The synthesized nanocomposite was characterized to confirm its EMI shielding effect, crystal structural, compositional and surface morphology. Our results are in whole agreement with already prepared material and the presence of each segment gave authorization about the composition in the next step we examine morphological and crystalline structure by using SEM and EDS. Which shows relevant results indicating confirmation of our desired values. In the final step we test our sample by using a network analyser to detect the shielding behaviour of polymer composite. The composite was tested with the reference of aluminium fibre of 6×6 length and then the material was coated over it with approximately 0.199mm of thickness. These coated samples were investigated from 2-18GHz in an anechoic chamber. And the results propose that shielding effectiveness of samples goes on increasing from 2-18GHz. According to above mentioned results and with the recently reported data our sample retains a modest shielding performance at merely a thickness of nearly 0.199mm. Further, the shielding may rise by rising the thickness of samples. Additional, the shielding seemed rise with the thickness of samples.

Acknowledgment

The authors would like to thank University of Engineering and Technology, Taxila for their support in the present work.

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