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AN EXPERIMENTAL EVALUATION AND TESTING OF CEMENTITIOUS BASED COMPOSITES WITH CONDUCTIVE ADDITIVES OF CARBONYL-IRON AND SILICA-FUME FOR THE EFFICIENT USE OF MINERAL WASTE FOR DEVELOPMENT OF COST-EFFECTIVE BROADBAND RADAR WAVE ABSORBER.

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ABSTRACT

The electromagnetic absorbing effectiveness of proposed radar absorbing based specimen with different contents involve: Carbonyl Iron as ferrite, conductive filler as carbon fibres/carbon black and admixture of silica fume with Portland cement studied in this paper. Double-layer Cementitious composites filled with Carbonyl Iron ferrite as microwave absorbers. The addition of silica fume, use to improve the impedance matching between the cementitious composites and free space. To design the Cementitious-based radar absorber, Carbon contents will be added to Carbonyl-iron and Silica-fume, since the study shows that these have high percentages of absorption values. This innovation material combination will be investigated to determine the best reflectivity performance of microwave absorbers. Carbon is the most important element that must be in the absorber to absorb unwanted microwave based material that has been used in Cementitious-based microwave absorber fabrication. Instead of using chemical and agriculture waste based material, this study shows that mineral waste is more effective and has much lower cost. The main objective is to achieve good absorption with wide bandwidth corresponds to reflection loss, RL ≤ -10 dB for absorber layer thickness about 10mm for cost-effective production of radar wave absorber. An experimental evaluation of this cement based composite is tested using dielectric probe method and radar cross-section method and therefore, reflection loss is tested under frequency range of 8 to 12GHz. A double layer approach is applied for obtaining good absorption. Carbonyl-iron and Silica-fume is an innovation in enhancing the microwave absorption properties of Cementitious-based radar absorber, to be used in radio frequency anechoic chambers. An anechoic chamber consists of radar absorbing material (RAM) along at its wall; floor and ceiling to eliminate unwanted reflections to create electromagnetically quite environment.With more and more severity of electromagnetic environment pollution, the study on building materials that can prevent electromagnetic interference(EMI) has caused great attention. So, The cement-based radar absorbing material used for EMI shielding and wave absorbing building materials.

Keywords*: *Carbonyl iron, Silica fume, Carbon fibres, Carbon black, ferrites, Impedance matching, Layered cementitious composites, Microwave absorption, Reflectivity.

I. INTRODUCTION

Now people are living in a more and more complicated electromagnetic environment. Actually, electrical devices have greatly improved the quality of our lives. However, everything has its bad effects. For example, sometimes we have to shield the electromagnetic radiations from such devices as computers, mobiles, and military devices to avoid leaking out of important information or avoid radar tracing. In other cases, the reflection of electromagnetic waves from the enclosure of high buildings can lead to the disorder of TV signals around the buildings. Now people are aware that radiation of electromagnetic waves may do harm to the health of human beings. Thus, development of building composite materials containing low cost components such as carbon black (CB) which are able to absorb or shield electromagnetic radiations becomes more and more necessary in the modern society.

The shielding effectiveness (SE) is the sum of three terms such as reflection loss, absorption loss and multi-reflections. So, SE is defined in decibels (dB) and its magnitude can be written as follows:

$$SE_T(dB)=10 \log (P_I/P_T) \qquad \text{eq. (1)}$$

where P_I and P_T are the electric fields that are incident on and transmitted through the shield. The reflectivity of -10 to -20 dB means that the incident electromagnetic waves have been reduced by about 70–90%. As the reflectivity of absorbing wave materials is less than -10 dB, they can be used in practice. Cement is slightly conductive, but its SE is very low. To increase the cement materials SE by adding a small amount of a conductive additive such as graphite powder, carbon black, carbon fibers, carbon filaments/steel fibers.

For the purpose of preparing a low-reflecting absorber in the desired wide frequency range, two fundamental conditions must be satisfied [7,8]: the first is that the incident wave can enter the absorber to the greatest extent (impedance matching characteristic),and the second is that the electromagnetic wave entering into the materials can be almost entirely attenuated and absorbed within the finite thickness of the material (attenuation characteristic).The impedance matching is the principle that the electromagnetic wave is absorbed in the materials. There are several methods to improve impedance matching between material and free space. One of them is to use low dielectric constant materials to adjust the characteristic impedance of the absorber. Silica fume [9] is a kind of fine non-crystalline silica produced in electric arc furnaces as a by-product used for this purpose.Carbonyl Iron has excellent absorptive abilities at lower frequencies and can widen frequency band when combined with other absorbents and having quality of good reflectors as it is used in the production of some ferrites.Typicaly applicable in radar absorbing material, EMI/RFI shielding product sand metal injection molded parts [9,10]. It’s well known that carbonyl-iron particles (CIP), which possesses excellent magnetic-loss property, in the frequency range of 2-18 GHz, is widely blended in polymer matrix as microwave absorbing materials [23-26].

So, Double-layered Cement based composite with involvement of such additives studied in this paper in which Silica-Fume present in Surface-layer and Carbonyl-Iron act as Loss-layer with carbon contents in order to increase its conductive value.

II. LITERATURE REVIEW

Guang HT, Liu SH, et al. [2007] and Wang C, Li KZ, Li HJ, et al. has presented cementitious composites are one of the most common building materials used in engineering construction. Cement-based composites are complex systems that include hydration products, unhydrated cement particles and aggregates of different sizes. Generally, as a whole system cement-based material is slightly conducting, but its EMI shielding effectiveness and wave absorbing property are very low, so admixtures are needed to improve the ability to resist the electromagnetic wave interference. There have been many studies on the reflection loss of cement matrix composites by introducing fillings, such as expanded Polystyrene (EPS) and carbon fibers [3,4].

Yamane T, Numata S, Mizumoto T, Naito Y. and Morimoto M, Kanda K, Hada H, et al. [2002] has studied ferrite is one of the most commonly used materials as a kind of electromagnetic wave absorber. Many studies have been carried out in Japan in Radio frequency (RF) area to investigate the electromagnetic absorption properties of buildings employing ferrite[5].

Cao MS, Zhu J, Yuan J, et al. [2002] shows the application has been restricted by the narrow band characteristics of single-absorbers. It is known from many research studies that the microwave absorber with double-layer structure has wider absorption bandwidth and lower reflection loss (RL) than the single-layer absorber in GHz frequency [6].

Zhang BS, Feng Y, Xiong J, et al. [2006] and Oikonomoua A [2007] studied for the purpose of preparing a low-reflecting absorber in the desired wide frequency range, two fundamental conditions must be satisfied [7,8]: the first is that the incident wave can enter the absorber to the greatest extent (impedance matching characteristic),and the second is that the electromagnetic wave entering into the materials can be almost entirely attenuated and absorbed within the finite thickness of the material (attenuation characteristic).The impedance matching is the principle that the electromagnetic wave is absorbed in the materials. There are several methods to improve impedance matching between material and free space. One of them is to use low dielectric constant materials to adjust the characteristic impedance of the absorber.

Toutanji Houssam A, et al. studied silica fume [9] is a kind of fine non-crystalline silica produced in electric arc furnaces as a by-product during the production of metallic silicon or ferrosilicon alloys, the SiO_2 content of which ranges from 85% to 98%.

Duan Yuping, et al. [2012] shows the microwave absorbing coatings with PVC (polyvinyl chloride) sheet as base plate are fabricated composed of CIP (carbonyl–iron particle) as absorbent and PU (polyurethane varnish) as matrix. The absorption properties of PVC-based coatings with different CIP content are investigated and compared with the corresponding Al (aluminium)-based coatings [10].

Luo X and Chung DDL. [2001] studied in order for a conductive filler to be highly effective for shielding, it preferably should have a small unit size, a high conductivity and a high aspect ratio. As to improving the conductive ability and shielding effectiveness of cement matrix composites, carbon fibres are more effective than particles such as carbon black and coke due to their large aspect ratio, which can help to make more conductive networks through intercalating [11–13].

Chung DDL. [2002] studied with the decrease in carbon fibre cost and the increase of demand for cement based composites with high structure and multi-function, carbon fibre cement matrix composites are gaining in importance quite rapidly.In the carbon fibre reinforced cement based composites,the carbon fibre with a diameter of more than 0.1 lm is often called fibre, whereas that with the diameter less than 0.1 lm is often called carbon filament. Due to its higher aspect ratio, carbon filament is superior to carbon fibre in shielding [14,15].

Fu X, Lu W, Chung DDL. [1996] explains when the carbon materials are used as the conductive fillers, it is necessary that the fillers be well dispersed, so it often needs to introduce some dispersants. Dispersants are not conductive themselves, but their introduction can obviously improve the dispersion degree of conductive fillers so as to help make more efficient conductive networks. Among the various types of dispersants, styrene butadiene latex and silica fume are the most common for use in cement based composites. Moreover due to the weak strength between the carbon fibre and cement matrix, the introduction of latex, silica fume or methylcellulose can improve the bond between the fibre and matrix, thereby improving the mechanical properties of the cement composites[16,17].

Chen PW, et al. [1995] studied the surface pretreatment of carbon fibre or treating silica fume with silane can improve the bond strength between carbon fibre and the cement matrix and the dispersion degree of conductive fillers, thereby increase the shielding effectiveness of the composites [18–20].

X. Zhang, et al. [2010] has studied the mortar with silica fume can be used as an impedance matching layer to adjust the permittivity of the surface materials of the cement-based absorbing material in order to attain the impedance matching.The microwave reflectivity of the single-layer mortar filled with ferrite is higher than that of the plain mortar due to the mismatching of the impedance and the design of double-layer structure has excellent absorption property because of the impedance match of materials. The impedance match layer is made of silica fume mortar and the loss layer is added with 30 wt.% ferrite based composite [21].

Dai Yawen, et al. [2010] has presented the filling of CB improves the loss factor of the cement material remarkably, which makes CBCC absorb electromagnetic waves by polarization. The loss factor of CBCC increases with the CB content increasing and the Compressive strength of CBCC decreases with CB content increasing. Compressive strength decreased substantially when CB content is more than 3 wt.% [22].

Main data drawn from Figure 1 and 2 is listed in Table 1. It can be found that the bandwidth in which the reflectivity is less than -10 dB decreased in the order: CBCC containing 0.5 wt.% of CB, CBCC containing 2.5 wt.% of CB, and 3.0 wt.% of CB. The absolute value of maximum reflectivity decreases in the order: CBCC containing 2.5 wt.% of CB, CBCC containing 0.5 wt.% of CB, and 3.0 wt.% of CB.

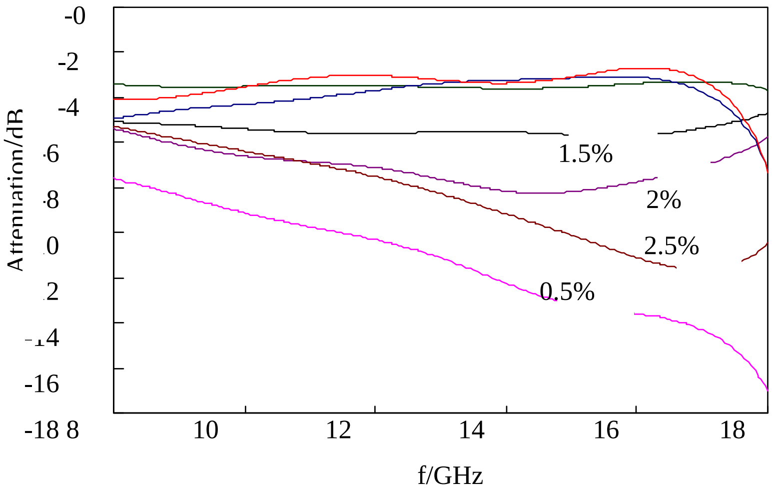


Figure. 1: The absorbing performance of CBCC with different concentration of CB in the frequency range of 8–18 GHz.

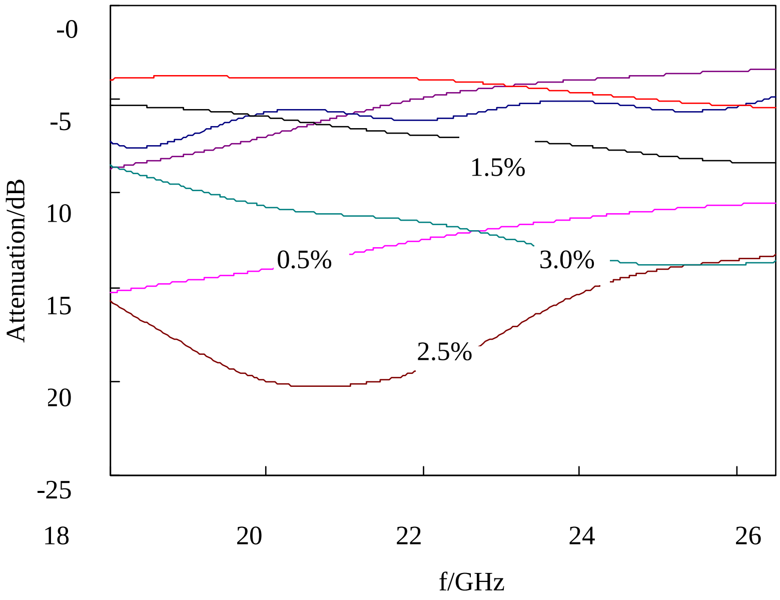


Figure. 2: The absorbing performance of CBCC with different concentration of CB in the frequency range of 8–26.5 GHz.

D. Yuping et al. [2012] studied that in order to characterize the microwave absorbing properties of the composite coatings based on Al or PVC sheet, the reflection loss (RL) curves versus frequency for different CIP content are simulated, and shown in Fig. 3. The content of CIP varies from 1:3 to 1:7 (PU:CIP mass ratio). The thicknesses of coating and base plate are 2 mm and 3 mm, respectively. The sweeping frequency ranges from 2 to 18 GHz. From Fig. 3(a), it can be found that the allowable reflection loss (RL ≤ -10 dB, for over 90% microwave absorption) can get in the frequency range of 5.5–13 GHz through varying the component content of the coating. It is worth noting that, in Fig. 3(b), the PVC-based coatings display good absorption properties in the lower frequency region (2–4 GHz, S-band), though the overall performance is poor compared with the Al-based coatings. Therefore,the microwave absorbing properties of composite coating based on Al and PVC studied in frequency range of 2-18 GHz.

Table. 1: Minimum reflectivity and bandwidth of CBCC in the range of 8–26.5 GHz.

Content of CB (wt%)	0.5	2.5	3.0
Minimum reflectivity (dB), at frequency (GHz)	-17.04 (18)	20.30 (20.6)	13.86 (25.3)
Bandwidth (reflectivity ≤ 10 dB, GHz)	11-26.5	14.9-26.5	19.2-26.5
Bandwidth (reflectivity ≤ 15 dB, GHz)	17.4-18.4	18-24.2	-

The results shows that the RL peaks of coatings shift towards the lower frequency region by increasing the CIP content or coating thickness.PVC-based coatings with a fixed component content of 1:7 (PU:CIP mass ratio) in CIP/PU layer, exhibit a minimum RL value of −29 dB at 4 GHz and a permissible RL (RL ≤−10 dB) band of 2–6 GHz through varying the thicknesses of PVC sheet and PU/CIP layer, which is much better than the performance of the common metal-based coatings in the lower frequency.

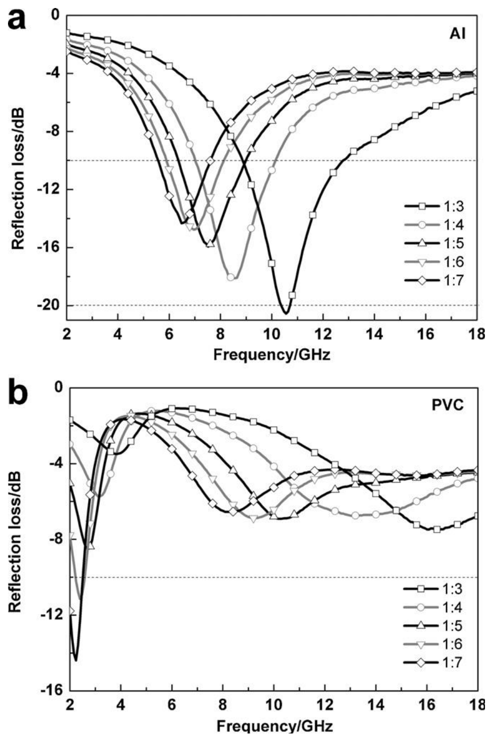


Figure. 3: Measured reflection loss curves versus frequency of the CIP/PU coatings with varied CIP content (PU:CIP mass ratio) based on Al (a) or PVC (b) sheet.

III. MATERIAL AND METHOD

3.1 Material

In this work, low cost mineral waste materials like Carbonyl-Iron powder is used as the microwave absorber in lower layer, as loss layer. The silica fume used as impedance matching and transmission line absorber in upper layer called as surface layer. The bonding agent used is cement and hardening agent used is sand. Carbon black/carbon fibers used in order to accelerate the rate of absorption. Napthelene powder used as water reducing agent. The specification and chemical composition of used materials are as follows:

Table. 2: Specification of Carbonyl-Iron

Atomic Number	Molecular Weight (g/mol.)	Density	Specific Heat	Boiling Point (^{0}F)	Melting Point (^{0}C)	Thermal Conductivity	Soluble in Water
26	195.9	7.87	12	217	1536	12	Insoluble

Table. 3: Chemical composition of Silica-Fume

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Loss	Specific surface (m^2/Kg)
95.48	0.27	0.83	0.54	0.97	0.80	1.11	22,000

Table. 4: Chemical composition of Portland cement, Grade-53

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Loss	Specific surface (m^2/Kg)
21.50	5.12	3.42	65.3	1.05	2.03	0.46	380

Table. 5: Specification of (a)Carbon-black & (b) Carbon-Fibres

Surface area (m^2/g)	pH scale value	Particle Size
1056	8.0	33 nm

(a)Carbon-black

Tensile strength (Gpa)	Density (g/cm^3)	Carbon Content (%)
≤3500	1.65-1.75	≤98

(b) Carbon-Fibres

3.2 Method

The exact ratio and four different Cementitious-based composites having dimensions of 20x20 cm^2 are prepared with different thickness discussed as below in Table 6 and 7.

Table. 6: Ratio of used material

S.No	Material	Description
1	Portland Cement	-
2	Carbon Black	5 wt% as that of cement
3	Silica Fume	15 wt% as that of cement
4	Carbonyl-Iron Powder	60 wt% as that of cement
5	Sand	1.5 : 1 (sand to binder)
6	Carbon fibers	5 wt% as that of cement
7	Napthalene Powder	0.5 wt% of cement
8	Al sheet (Grade- 6061)	5mm (thick)

Table. 7: Thickness and Materials used in each sample

S.No	Mixture (Layer)	Thickness
1	C+S+CB+SF+N (Single)	10 mm
2	C+S+CF+CI+N (Single)	10 mm
3	C+S+CB+SF+N C+S+CF+CI+N (Double)	1st layer- 2 mm 2nd layer- 2 mm
4	C+S+CB+SF+N C+S+CF+CI+N (Double)	1st layer- 2 mm 2nd layer- 4 mm

Where, C=Cement, S=Sand, CI=Carbonyl-Iron, SF=Silica-Fume, N=Napthalene, CB=Carbon-black, CF=Carbon-Fibres.

The preparation of samples in detail as follows:

Single-layer:

1st Sample- Mix all the materials properly given in Table 8. Add water in proportional to the cement in order to make a thick paste, then pour the paste into the 10mm thick plywood mould and place the mould on the vibrator for 30 seconds, to remove the air bubbles and to get settle down the mixture in all corners with the help of vibrator. Then Place the mould at room temperature for 24 hours to get solidified. After that demoulded it and put the sample in curing process for 28 days. The water level under curing process must be little above from the sample. After curing, In order to reduce the influence of free water in composite material, sample dried at 60^0 C prior to test with the help of Thermostatic Oven, so that its weight do not change with time. Place the fabricated single cementitious-based layer on the Aluminum sheet.

Figure. 4: Vibrator

Figure. 5: Curing

Figure. 6: Oven

2nd Sample- The preparation of second sample same as that of first.

Double layer:

3rd sample- Firstly make loss layer, Then pour the paste of surface layer onto loss layer, rest all procedure is same as previous one.

4TH sample- All the procedure is same as that of 3rd sample.

Figure. 7: Fabricated cement based samples

Figure. 8: double layer view

Figure. 9: CI

Figure. 10: CB

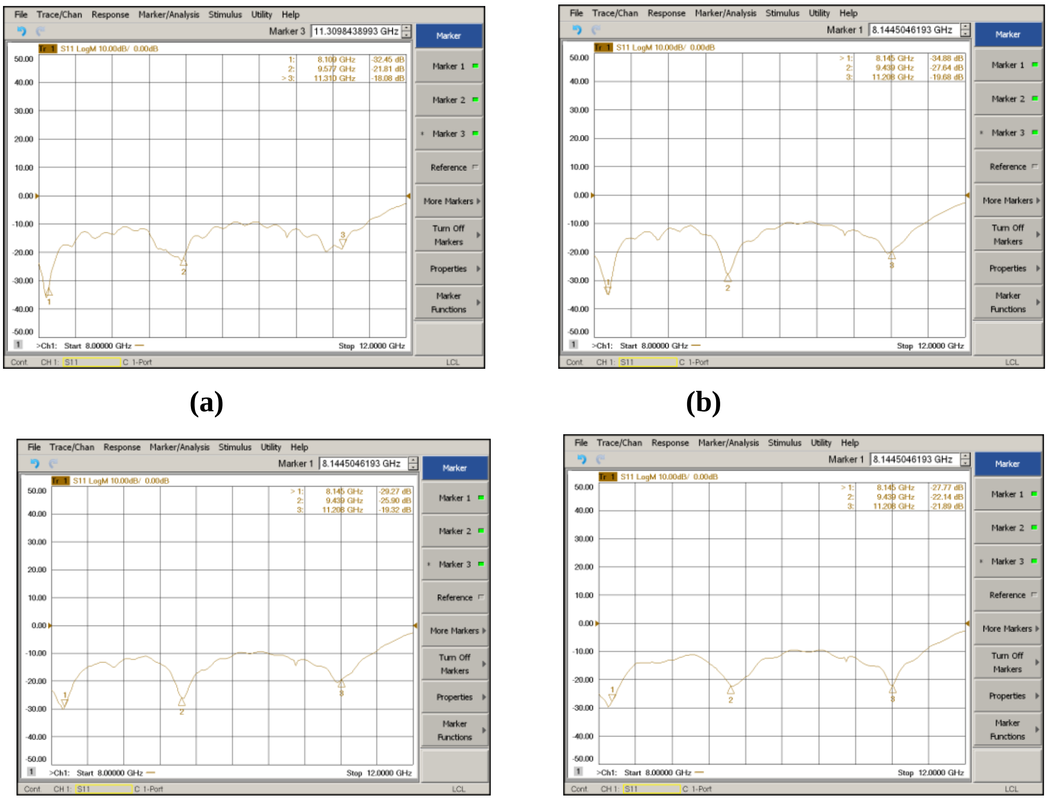
Figure. 11: CF

Figure. 12: SF

IV. TEST METHOD WITH RESULTS AND DISCUSSION:

4.1 Vector Network Analyser (VNA)- The Vector Network Analyser is the most important instrument in microwave measurement. Its primary function is measurement of S-parameters. It calculates both magnitude and phase thus this is called Vector Network Analyser. Both Reflection and Transmission characteristics are measured by Network Analyser. Network Analysis is concerned with the accurate measurement of the ratios of the reflected signal to the incident signal, and the transmitted signal to the incident signal. The used VNA operating in the range of 10MHz-67GHz and far field taken as 21 cm. The frequency range chosen for the measurement is 8 to 12 GHz for. Horn Antenna and a co-axial cable is used for the testing process. Horn Antenna is connected with the co-axial cable and the co-axial cable is connected with the port 1 of VNA. Calibrate the VNA using aluminium sheet. Calibration is done in order to make aluminium sheet showing maximum reflection that is no absorption. Place the MUT (Material under Test) exactly in front of the Horn Antenna one by one at the distance of 30cm, so that maximum reflections must be observed by Horn Antenna.

The obtained S-parameters graphs are shown in below figures:



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Marker 3  11.3098438993 GHz
  1:     8.109 GHz   -32.45 dB
  2:     9.577 GHz   -21.81 dB
> 3:    11.310 GHz   -18.08 dB
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(a)

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Marker 1  8.1445046193 GHz
> 1:     8.145 GHz   -34.88 dB
  2:     9.439 GHz   -27.64 dB
  3:    11.208 GHz   -19.68 dB
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(b)

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Marker 1  8.1445046193 GHz
> 1:     8.145 GHz   -29.27 dB
  2:     9.439 GHz   -25.90 dB
  3:    11.208 GHz   -19.32 dB
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(c)

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Marker 1  8.1445046193 GHz
> 1:     8.145 GHz   -27.77 dB
  2:     9.439 GHz   -22.14 dB
  3:    11.208 GHz   -21.89 dB
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(d)

Figure. 13: (a) S-parameter graph of 1st sample (b) S-parameter graph of 2nd sample (c) S-parameter graph of 3rd sample (d) S-parameter graph of 4th sample

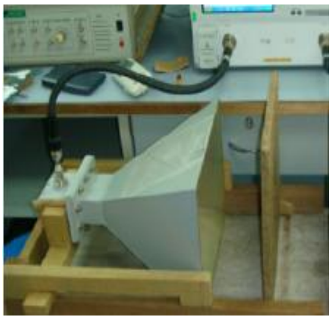

Figure. 14: Sample in front of Horn Antenna

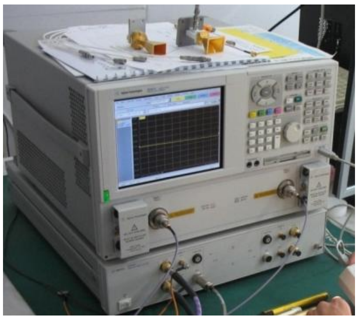

Figure. 15: VNA

4.2 Dielectric Probe Method- Another common use of VNA is measurement of permittivity and permeability of the materials. The method used for the calculation of dielectric constant is called Dielectric Probe Method. In this method, two co-axial cables are used to calculate the dielectric conctant of MUT. These are designed to calculate dielectric constant in the range of 8 to 12 GHz. The method works best for the sample loaded in waveguide, although transverse electromagnetic guide such as co-axial line can also be used. The sample has to be in the shape that fit exactly inside the waveguide. Before measurement, calibration of VNA is required. The results obtained from this method by using 85071 Material Measurement Software E07.01.08 through Agilent Technologies N5247A.

(a) **(b)** **(c)**

Figure. 16: (a) X281C Adapter (b) WR-90 Waveguide size sample (c) MUT inside waveguide

V. CONCLUSION

Microwave absorption, reflectivity and dielectric constant properties of single-layer and double-layer cementitious based radar absorbent containing different contents of silica fume and carbonyl iron as absorbers with involvement of carbon particles have been studied in this paper. The conclusions can be summarized as follows:

(1) The mortar with silica fume can be used as an impedance matching layer to adjust the permittivity of the surface materials of cement-based absorbing material in order to attain the impedance matching.

(2) The microwave absorption of single layer is more than that of double layer cement based composite attaining close to -38 dB as shown in Figure. 13 (a) and (b), which put limitation to double layer cement based radar absorbent, investigated in this work.

(3) The filling of carbon contents to the basic material improves the loss factor of cement material remarkably, which makes cement based composites absorb electromagnetic waves by polarization. The loss factor of cement based radar absorbent increases with carbon content increasing.

(4) The reflection loss of double-layer plate reaches -29 dB at 8.1 to 8.12 GHz and -25 dB at 9.42 to 9.48 GHz. For single-layer reaches -36 dB at 8.08 to 8.1 GHz and -35 dB at 8.14 to 8.16 GHz with thickness of specimens about 10 mm and absorption bandwidth below -10 dB for both single and double-layer cement based radar absorbent composites.

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