A Review on Epoxy Composites using Hollow Glass Microspheres and Nanoreinforcements

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ABSTRACT

Epoxy is a key structural material for marine, automotive and aerospace applications. It is well known that epoxy resins are brittle and poor in mechanical and thermal properties. Introducing micro and nanoscale reinforcements to the epoxy is a potential approach to achieve enhanced mechanical and thermal properties. To date, much work has been done on hollow glass microsphere (HGM) reinforced epoxy composite. However, few systematic studies about the influence of the nanoreinforcements of the mechanical and thermal properties on HGM/epoxy composites were conducted. These composites and hybrid nanocomposites constitute an alternative product to metal-based ones and show great potential as multifunctional materials for a wide variety of applications, such as civil construction, automotive, aerospace, optoelectronic devices, semiconductor devices and others. This paper presents a systematic review of the different properties of HGM/epoxy composites. Moreover, we also review that mechanical and thermal properties of HGM/epoxy is enhanced by introducing nanoreinforcements.

KEY WORDS: Nanoreinforcement, Hollow Glass Microsphere, Epoxy, Tensile Strength, Compressive Strength.

1. INTRODUCTION

Epoxy have attractive properties like good mechanical properties, excellent adhesion and good chemical and heat resistance, making them useful for automotive, industrial, defense and aerospace applications (Yung,2009; Kim, 2001; Huang, 2012; Keivani, 2015; Wang, 2010). However, epoxy resins are brittle in nature and show a large coefficient of thermal expansion, poor compressive strength, which limits their applications. Additionally, for aircraft and aerospace applications, epoxy resins are required to be light weight, low thermal conductivity.

Presently, few groups were developed hollow glass microsphere (HGM)/epoxy composite, also known as syntactic foam (Ferreira, 2010; Koopman, 2004; Choquese, 2010; Mutua, 2012; Yagoubi, 2012), as materials for automobile, marine and aerospace applications. It has higher excellent specific compressive strength, low moisture absorption, high dimensional stability and good thermal insulation properties. The advantages of HGM/epoxy composite foam over epoxy composites are lighter weight materials and materials with similar structural dimensions, but superior properties, such as very high compressive strength, high dielectric constant, high chemical resistance, high electrical insulator etc. Yet, as the use of composite foam materials in modern applications is increasing, these materials are now extensively used in marine, automobile and aerospace applications.

Consequently, epoxy composite foams are reaching their limit. Introducing nano-materials into the HGM/epoxy is a potential solution to improve the mechanical, thermal, dielectric and thermomechanical properties. Few groups (Shutov, 1986; Rutz, 2012; Samsudin, 2011; Gupta, 2010; Moynot, 2006), modified HGM/epoxy foam by introducing nanoreinforcements (nanoR) like nanoclay (NC), graphene (GPN), carbon nanofiber (CNF). They found that nano based HGM/epoxy foams are thermally stable and their compressive strength and dielectric constants are greatly improved (Gupta, 2002; 2003; Thomas, 2009). It is observed that the addition of nanomaterials not only increases the strength of the epoxy matrix but also serves as a mechanism to contain the microcracks from developing into macro cracks. On the basis of research results, it can be said that nanocomposite foam plays an important role in packaging industries and also for short-term disposal applications. The first part of this review focuses on different properties of HGM/epoxy syntactic foam. In the second part of this review, nanomaterials for different applications are introduced into the HGM/epoxy composites. We also introduced the various reinforcement strategies used with these nanomaterials.

Hollow Glass Microsphere and Nanoreinforcemnts: Currently, a wide range of HGM is made with different commercial names. Hollow glass microspheres deliver the benefit of high heat and chemical resistance, but with the added property of lighter weight. Fig. 1 shows the SEM micrograph of HGM. Spheres with smooth surfaces and average diameters of between 10 and 30 µm are the common morphological characteristics of these HGMs.

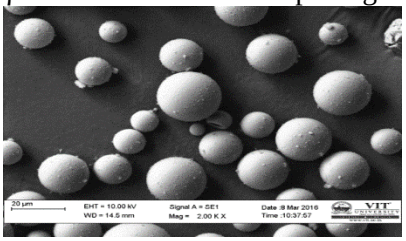

Fig.1. SEM image of HGM

It is extensively used in industrial areas ranging from sealants (Ronald, 2013), reinforced plastics (Yu, 2013), offshore products (Song, 2005), and coatings (Song, 2005) etc. The nanoscale fillers considered are carbon nanotubes (CNTs), carbon nanofiber (CNF) (Viot, 2008), graphene (GPN) (Shams, 2013), and nanoclay (NC) (Wouterson, 2005) etc .

HGM/epoxy composites and nanoR/HGM/epoxy composites: Nanomaterials are considered to be an excellent candidate as a reinforcing phase for various composite materials, owing to its excellent mechanical, thermal and electrical properties. They have large surface area, which makes them very active and forms bonding between a nanomaterials and a matrix material for fabricating composite material. Various types of nanoR such as CNT, CNF, NC, GPN etc. have been verified for their abilities to enhance the mechanical and thermal behaviors of HGM/epoxy.

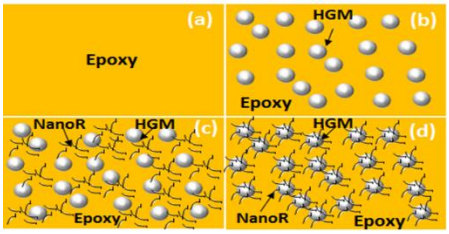

Fig.2. Diagram of the (a) epoxy, (b) resulting HGM/epoxy composites and (c &d) resulting nanoreinforcement/HGM/epoxy composites

They have been less studied in the framework of nano-reinforced HGM/epoxy composites. More reviews have been published by Gupta (2002; 2003). The diagram of the resulting HGM/epoxy composites and nanoreinforced syntactic foams is shown in Fig. 2.

Processing of HGM/epoxy and nanoreinforced HGM/epoxy composites:

Preparation: As reported by Yung (2009) and Kim (2001), the HGM powder was pre-treated by a coupling agent usually KH560 (silane) to improve its performance. Then HGM in required ratio is mixed with epoxy resin and stirred well by a high speed mixing for hours to obtain a homogeneous blend else the HGMs would have float on the surface of the epoxy due to its low density. The polymer preparation is carried out in two steps, initially by heating the mixture to a temperature of 80-145^{o}C, simultaneously it is pumped for an hour or more to remove solvents and air bubbles and then the polymerization process is completed by heating the mixture to 175^{o}C for 4 hours. The mixture is then poured in mould and cured for 12 hours under 0.016 MPa pressure and high temperature.

In the nanoreinforcement composites, the nanomaterials are introduced in two ways: (a) in the matrix (Ronald, 2013); (b) growing/depositing nanomaterials over the HGM surface (Zegeye, 2012), as shown in Fig. 3. To get nano reinforced composites, initially the nano particles are mixed in epoxy resin either by a shear impeller fitted to mechanical mixer or by ultrasonication.

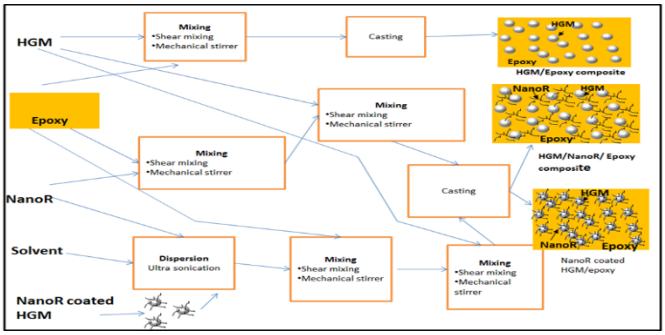

Fig.3. Diagram of processing techniques of HGM/epoxy and nanomaterials/HGM/epoxy composites

Then HGMs are added to the nano/epoxy mixture and the slurry is mixed for additional 15 minutes. Then the entire mixture has been degassed followed by usual casting and curing. Agglomeration of nanomaterials is a challenge in the preparation of composites, thus in some work as in[33], the nanomaterials are mixed in epoxy by solvent deposition technique, where the nano-reinforcement are mixed with some solvents as toluene and the solution is mixed in epoxy followed the ultrasonication or mechanical mixing and the addition of HGMs. The composite is obtained by casting followed by curing. Zegeye (2012), reported the CNTs grown over the HGM surface by CVD method. These CNTs coated HGM are mixed into the epoxy and are cast to prepare composites.

Processing Challenges: The processing of HGM/epoxy composites is very difficult by poor solubility of HGMs. Because of size, HGM will tend to agglomerate when dispersed in an epoxy resin. Scientists are using many functionalizing agents to achieve HGM solubility within the epoxy matrix. Methods used include functionalizing the HGM with functional groups that serve in uniformly dispersing the HGM in the monomer, dispersing the HGM in the monomer with smallest processing of the HGM, or adding surfactants or polymers that coat individual HGM and prevent the HGM from coagulation. Nanomaterials have also poor solubility in many solvents and most liquids such as water, polymer resins etc. Due to the poor solubility of nanomaterials in water, polymer or organic solvents, they are very difficult to isolate one nanomaterial from the other. When nanomaterials are blended with polymers for

preparing composites the dispersion property of nanomaterials has become more important[34]. The big challenges encountered in making such a composite are the uniform dispersion of nanomaterials in epoxy matrix without agglomerates and entanglement. Due to the atomically non-reactive surface of nanomaterials, lack of interfacial bonding limits load transfer from the matrix to the nanotubes. These problems can be overcome by functionalization of the nanomaterials with such groups that will form some kind of bonds with the epoxy matrix phase, such as vander waals bonds, hydrogen bonds and chemical bonds etc. (Colloca, 2013; Gupta, 2013).

Mechanical properties nanoR/HGM/epoxy composites: The mechanical response of epoxy materials strongly depends on the HGMs and nanomaterials content and is generally characterized with respect to different properties, such as the tensile strength, compressive strength, flexural strength, fracture toughness, etc.

Tensile properties: In general, tensile properties were determined as per ASTM D638. Table.1 summarizes the tensile properties of HGM/ epoxy and HGM/nanoR/epoxy composites reported in the literature.

Table.1.Modification of tensile properties of epoxy by HGM and nanoreinforcements

Ref.	HGM content (vol. %)	NanoR (vol. %)	T.S. (var. %)	T. M. (var. %)
Shutov, 1986	10	-	-0.8	-
Shutov, 1986	20	-	-2.0	-
Shutov, 1986	30	-	-4.6	-
Gupta, 2010	30	-	-	22
Gupta, 2010	40	-	-	37.8
Asif, 2010	39	1 (NC)	12.87	51.49
Asif, 2010	37	3 (NC)	12.87	44.02
Asif, 2010	35	5 (NC)	6.07	47.76
Asif, 2010	40	1 (PEEKMOH)	26.19	51.49
Asif, 2010	40	3 (PEEKMOH)	19.49	51.49
Asif, 2010	40	5 (PEEKMOH)	26.19	51.49
Zegeye, 2014	30	0.1 (GPN)	6.07	55.22
Zegeye, 2014	30	0.3 (GPN)	6.07	51.4
Colloca, 2013	30	0.42 (CNF)	45.58	17.91
Colloca, 2013	50	0.3 (CNF)	46.25	22.01
T.S.: Tensile strength; T.M.: Tensile Modulus; Var.: Variation, NanoR: Nanoreinforcement				

The tensile strength of the epoxy increased with a decrease of the volume fraction of HGM (Shutov, 1986). Research work by Asif (2010), shows that the specific tensile properties of the epoxy syntactic foam with PEEKMOH or nanoclay and PEEKMOH toughened epoxy clay syntactic foam are presented in Fig. 4. The specific tensile strength was improved by 13% by introducing 1 wt% NC, afterward it decreases with increase in clay content. The specific tensile modulus was also improved at 3 wt% clay and thereafter decreases.

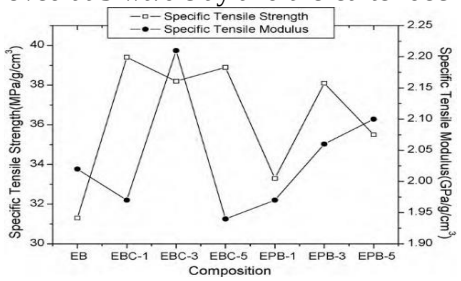

Fig.4. Specific tensile strength and modulus versus composition of epoxy syntactic foam with nanoclay or Hydroxyl terminated poly ether ketone (PEEKMOH) (Asif, 2010)

According to Colloca (2013), it added CNF into epoxy composites to test its tensile properties. The results show increase in tensile strength and tensile modulus of CNF reinforced epoxy composites up to certain extent. Table.1 also presents a comparison between tensile modulus and strength of various epoxy syntactic foams, with and without CNFs.

Compressive properties: Generally, the compressive properties of composites are enhanced by adding HGMs and nanoR to epoxy matrix as shown in Table.2 (Ronald, 2013; Wouterson, 2005; Asif, 2010; Zegeye, 2014; Poveda, 2014). According to experimentation conducted by Wouterson (2005) and Zegeye (2014), it shows that HGM and nanoR in the epoxy changed the stress strain patterns (Fig.5a and b). In Fig. 5a, they have identified three regions in the curves. The first region is illustrated by a nearly linear-elastic behavior of the epoxy syntactic foam. The compressive strength of syntactic foam is found out the ends of the first region. The second region of the curve is attributed to the crumbling of the HGM under the increasing compression load. The third region is reflected by a sharp increase in the load–displacement curve and the sharp increase is produced by a large number of HGM being crushed and compacted, and reaching at the maximum density. In Fig.5b, similar compressive stress–strain profile

was observed. Reference[32] shows that enhancement of compressive strength and compressive modulus, were achieved by 5 wt % NC and 5 wt% PEEKMOH.

Table.2.Modification of compressive properties of epoxy by HGM and nanoreinforcements

Ref.	HGM content (vol %)	NanoR (vol %)	C.S. (var. %)	C. M. (var. %)
Ronald, 2013	30	-	-12.32	15.38
Ronald, 2013	50	-	-41.54	15.38
Ronald, 2013	30	-	34.44	87.4
Ronald, 2013	50	-	22.75	44.23
Wouterson, 2005	10	-	-38.09	-15.38
Wouterson, 2005	20	-	-36.66	-34.61
Wouterson, 2005	30	-	-47.70	-36.53
Wouterson, 2005	40	-	-55.62	-39.42
Wouterson, 2005	50	-	-63.56	-39.42
Asif, 2010	40	-	-43.88	3.84
Asif, 2010	39	1(NC)	-27.51	11.53
Asif, 2010	37	3(NC)	-21.67	10.57
Asif, 2010	35	5(NC)	-16.99	14.42
Asif, 2010	40	1(PEEKMOH)	-24.01	13.46
Asif, 2010	40	3(PEEKMOH)	-26.35	11.53
Asif, 2010	40	5(PEEKMOH)	-18.16	14.42
Asif, 2010	39	5(PEEKMOH), 1(NC)	-16.99	12.5
Asif, 2010	37	5(PEEKMOH), 3(NC)	-5.30	20.19
Asif, 2010	35	5(PEEKMOH),1(NC)	-8.81	18.26
Asif, 2010	37	1(PEEKMOH), 3(NC)	-19.33	12.5
Asif, 2010	37	3(PEEKMOH), 3(NC)	-11.15	16.34
C.S.: Compressive strength; C.M.: Compressive Modulus; Var.: Variation, NanoR: Nanoreinforcement				

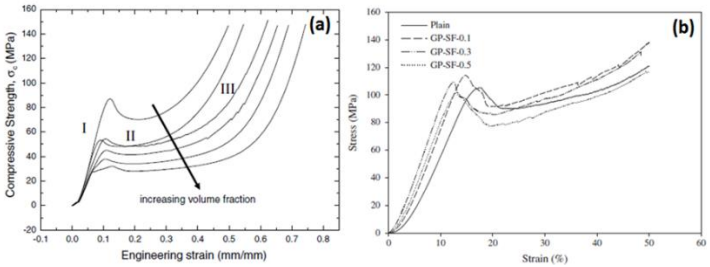

Fig. 5: (a) Compression stress–strain curves of epoxy syntactic foam with various amounts of HGM content, (b) Typical compressive stress–strain curves of various epoxy syntactic foam specimens

The enhanced compressive properties of the hybrid (NC/PEEKMOH) syntactic foams are due to the improvement of the surface area of interaction between NC layers and epoxy matrix as well as the toughening effect of PEEKMOH to the epoxy matrix. Research work by Zegeye (2012), shows that the compressive modulus is increased with increasing GPN content from 0.1 to 0.3 vol. %. The compressive strength also increases with increasing of low vol. % and decreases with addition of high vol. % of GPN.

Flexural properties: The flexural properties of a material by measuring the deflection of a sample under applied load is perform by the three-point bending test. Figure.6a show a decreasing trend in the maximum flexural strength with increasing HGM content. Similar to the tensile test results, the specific strength approaches a minimum around 40–50 vol. % of filler content. Table 3 lists the flexural strength of HGM/epoxy and nanoR/HGM/epoxy composites.

Asif (2010), reported that the flexural strength of the epoxy syntactic foam were increased with increasing NC, PEEKMOH and hybrid (NC/PEEKMOH) nano reinforcement, as shown in Fig. 6(b). For 5 % of NC, flexural strength is increased by 65%, whereas, fletural strength are increased by 57 % for 3 % PEEKMOH. This is because of the interaction between epoxy matrix and NC, PEEKMOH, hybrid nano reinforcement.

Table.3. Modification of flexural strength of epoxy by HGM and nanoreinforcements

Ref.	HGM content (vol. %)	NanoR (vol. %)	F.S. (var. %)
Ferreira, 2010	10	-	-1.35
Ferreira, 2010	26	-	9.95
Ferreira, 2010	43	-	2.71
Ferreira, 2010	50	-	17.19
Wouterson, 2005	10	-	34.38
Wouterson, 2005	20	-	41.62
Wouterson, 2005	30	-	45.70
Wouterson, 2005	40	-	61.99
Wouterson, 2005	50	-	74.66
Asif, 2010	39	1(NC)	52.03
Asif, 2010	37	3(NC)	57.91
Asif, 2010	35	5(NC)	65.61
Asif, 2010	40	1(PEEKMOH)	44.79
Asif, 2010	40	3(PEEKMOH)	57.01
Asif, 2010	40	5(PEEKMOH)	40.72
Asif, 2010	39	5(PEEKMOH), 1(NC)	59.72
Asif, 2010	37	5(PEEKMOH), 3(NC)	68.32
Asif, 2010	35	5(PEEKMOH),1(NC)	72.39
Asif, 2010	37	1(PEEKMOH), 3(NC)	66.96
Asif, 2010	37	3(PEEKMOH), 3(NC)	65.15
F.S.: Flexural strength; Var.: Variation, NanoR: Nano reinforcement			

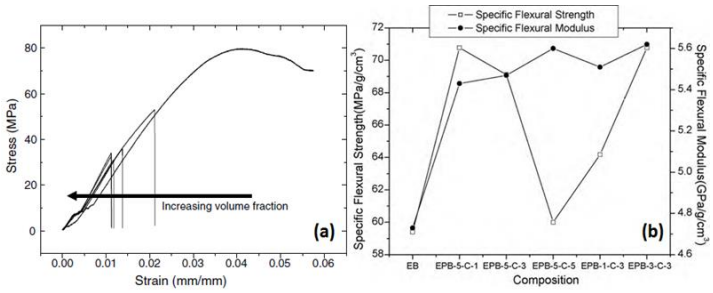

Fig.6. (a) Flexural stress–strain curves of epoxy syntactic foam with various amounts of HGM content 25, (b) Specific flexural strength and modulus versus composition of epoxy syntactic foam with nanoclay or PEEKMOH32

Fracture Toughness: The fracture toughness test is performed to find the toughness of a material in terms of the critical stress intensity factor (K_{IC}), and the critical strain energy release rate (G_{IC}), at the fracture initiation. The experimental results for the fracture toughness of the HGM/epoxy and nanoR/HGM/epoxy composite are listed in Table 4 (Wouterson, 2005; Asif, 2010).

Table.4. Modification of fracture toughness of epoxy by HGM and nanoreinforcements

Ref.	HGM content (vol. %)	NanoR (vol. %)	F.T. (var. %)
Wouterson, 2005	10	-	14.45
Wouterson, 2005	20	-	44.5
Wouterson, 2005	30	-	39.75
Wouterson, 2005	40	-	13.25
Wouterson, 2005	50	-	-14.45
Asif, 2010	39	1(NC)	2.4
Asif, 2010	37	3(NC)	2.4
Asif, 2010	35	5(NC)	4.8
Asif, 2010	40	1(PEEKMOH)	19.27
Asif, 2010	40	3(PEEKMOH)	1.2
F.T.: Fracture Toughness; Var.: Variation, NanoR: Nanoreinforcement			

Fig.7a illustrated the fracture toughness of epoxy composited with variation of HGM content. Introduction of 5% NC in epoxy syntactic foam is increased the fracture toughness, whereas, more enhancement of fracture toughness of the epoxy syntactic foam by introducing 1 % PEEKMOH.

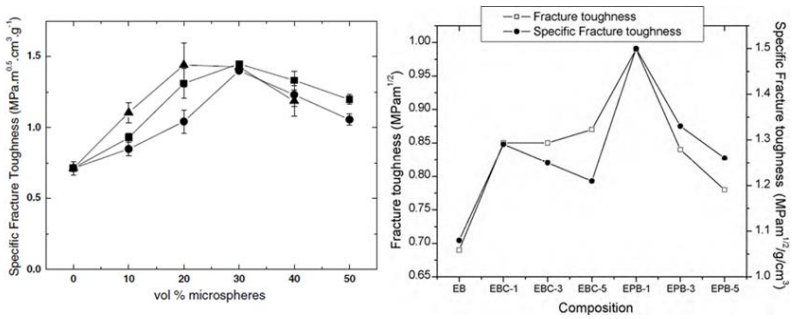

Fig.7: (a) Specific fracture toughness vs. filler content for syntactic foam25, (b) Fracture toughness and specific fracture toughness versus composition of epoxy syntactic foam with clay or PEEKMOH32

We can say that epoxy syntactic foam has lower fracture toughness than nanoreinforced epoxy syntactic foam. This is due to the synergic effect of interaction between NC, PEEKMOH and epoxy matrix.

Dielectric properties of nanoR/HGM/epoxy composites: The dielectric constant (D_k) and dissipation factor (D_f) of epoxy and their variations with increasing HGMs and nanoR/HGM are shown in Fig. 8(a) and Fig. 8(b), respectively. In Fig. 8(a), it is seen that the D_k is decreased with increasing HGM content. The D_k of epoxy syntactic foam (HGM-52%) shows maximum decrease of 41% compared with neat epoxy. Fig. 8(a) is also shown the D_f of the epoxy syntactic foam composite as a function of the HGM content (Yung, 2009; Poveda, 2014; Shadlou, 2014). It can be found that the D_f decreases with increasing HGM content, which is due to the low D_f for HGM (Table 5).

Table.5.Modification of dielectric properties of epoxy by HGM and nanoreinforcements

Ref.	HGM content (vol. %)	NanoR (vol. %)	D_f (var. %)	D_k (var. %)
Yung, 2009	10 vol % HGM	-	-13.88	-8.56
Yung, 2009	20 vol % HGM	-	-22.22	-13.85
Yung, 2009	33 vol % HGM	-	-30.55	-23.17
Yung, 2009	45 vol % HGM	-	-38.88	-26.95
Yung, 2009	52 vol % HGM	-	-41.66	-28.46
Poveda, 2014	15 vol% HGM	1(CNF	-87.7	64.58
Poveda, 2014	15 vol% HGM	2 wt% CNF	-87.5	100.41
Poveda, 2014	15 vol% HGM	5 wt% CNF	-88.2	222.91
Poveda, 2014	15 vol% HGM	10 wt% CNF	-86.8	300
Poveda, 2014	30 vol% HGM	1 wt% CNF	-88.4	60.41
Df: Dissipation factor; Dk: Dielectric constant; Var.: Variation, NanoR: Nanoreinforcement				

Poved (2014), reported that CNF/HGM/ epoxy have higher dielectric constant than the values for neat epoxy, as shown in Fig.8b. There was a noticeable high dielectric constant found for specimens containing 10 % CNFs.

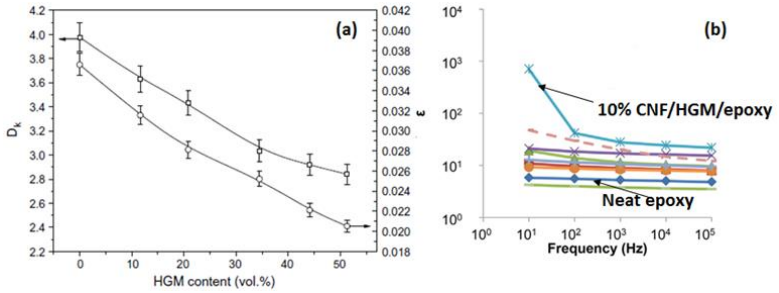

Fig. 8: (a) Dielectric constant (Dk) and dissipation factor (Df) as a function of HGM content at the1, (b) Comparison graph of dielectric constant of 220-type CNF/syntactic foam composites (Poveda, 2014)

2. CONCLUSION

In this review, we reviewed the preparation and different properties of nanoR/HGM/epoxy composites. Homogenous dispersion of nanomaterials and HGM in the epoxy is very important to enhance the properties of epoxy composites. The mechanical and dielectric properties of the epoxy composites were significantly changed with the addition of HGMs and a small amount of nanomaterials. Nanoreinforcement/HGM-reinforced epoxy composites as high-performance materials are currently of great interest for use in a wide range of marine, aerospace, and automative applications.

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