

Effect of Silica-Based Wastes on Wear Rate and Hardness Properties of Epoxy Composites as a Construction Material

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Abstract. In this study, polymer composites were manufactured with epoxy-based resin and wastes as a mineral additive. The wastes including a high content of silica (Silica fume, glass and fly ash) powder were used as fillers for an epoxy adhesive to improve its wear resistance properties. They were supplemented to mixes in various ratios via substituting the resin from 0 to 20% by weight. Tests of wear rate and hardness were conducted upon all-polymer composites at all fillers ratios. Results indicated that the epoxy hardness increased with increasing the filler addition. Consequently, the addition of wastes that include silica raised the wear resistance of polymer composites; nevertheless, it caused the composites harder materials. The wear rate decreased with increasing the silica fume, glass, and fly ash addition. In the case of fly ash addition, the minimum wear rate was at 15%, and after this percentage, the wear rate increased. However, in the case of glass addition, the minimum wear rate was at 10%, and after this percentage, the wear rate increased.

Introduction

Composites have been broadly utilized in engineering uses owing to their high strength-to-weight ratios [1]. Presently, the polymer composites combine unique characteristics; high deformation and strength properties, and concurrently, the low specific weight being extensively implemented in the industry [2]. The particle fraction and size influence on the mechanical and wear behaviors of polymeric composites was studied, and it was reported that the abrasion withstanding of composite reduces with the increment of the particle volume percentage, instead of an associated increase in the modulus of elasticity and hardness. The particle reinforced polymer composites can be used in many applications due to their low cost, isotropic properties, and easy manufacturing process [3].

Epoxy resins being broadly utilized as a matrix in numerous reinforced composites; they are a thermoset materials class of specific attention to the structural engineers due to the truth that these materials offer a sole balance of the mechanical and chemical characteristics that combined with an extensive processing versatility [4–7]. The epoxy resin possesses a vigorous cohesive force effect due to the presence of the Ester and Ether keys and the epoxy resin. Also, the epoxy resin possesses a vigorous cohesion of the molecular structure compact, thus the mechanical characteristics of the epoxy resin being better than the unsaturated phenolic resin and poly vinegar [8]. Some kinds of stiff inorganic particulate fillers have been investigated for improving the mechanical characteristics of epoxy composite, like alumina, nano clay, silica, etc. Among many epoxy fillers, Silica is the highest one utilized commercially as filler [9,10].

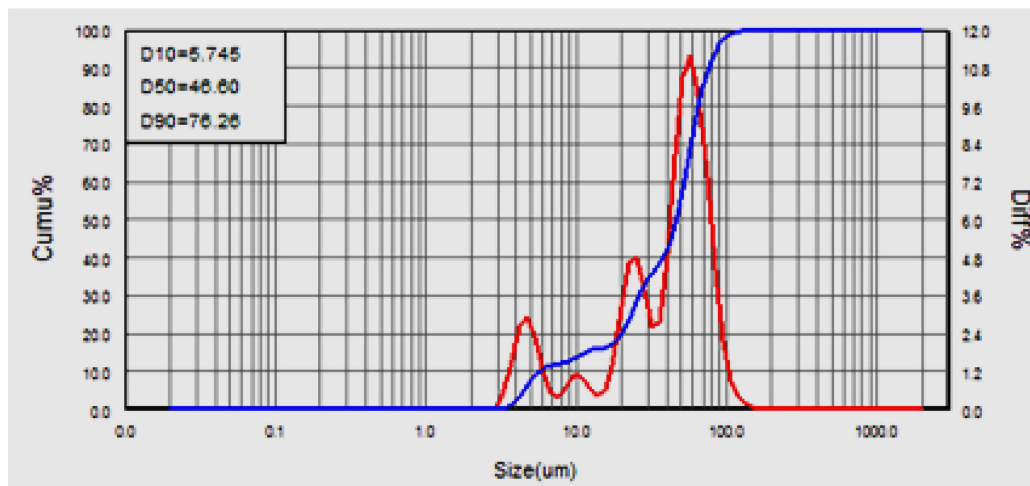
The particles of silica are normally utilized for enhancing the stiffness of epoxy owing to their high elastic modulus and vigorous adhesion with the matrix of epoxy. This adhesion limits the molecular mobility and the deformation of the matrix of epoxy at the interface area. The strengthened interface area enhances the transfer of load and the epoxy/silica stiffness [11]. Due to the incorporation of micro silica into the matrix of epoxy, the thermal and mechanical characteristics, as well as the dimensional stability of the neat epoxy regime, were enhanced through its method of

production or the life in service [12,13]. The epoxy resin-based materials cost being nearly more owing to the need and the consumption of energy. If the saving of energy and preservation of the natural resources being regarded, the substituted constituents use like the waste in the building materials being nowadays a global concern [5,14]. Thus, there is a need to find a silica source to decrease the costs of manufacturing production costs and acquire an encouraging impression for the surroundings (owing to the waste decrement) [15]. The use of industrial and other wastes in various applications as a building material has been implemented in many of the previous works [16–21]. Besides silica fume [22,23], one of the by-products commonly used as a building material in various applications is fly ash [24–26]. So it was used in this study as a source of silica. Also, glass wastes [27] are also widely used as a construction material, which is mainly composed of silica.

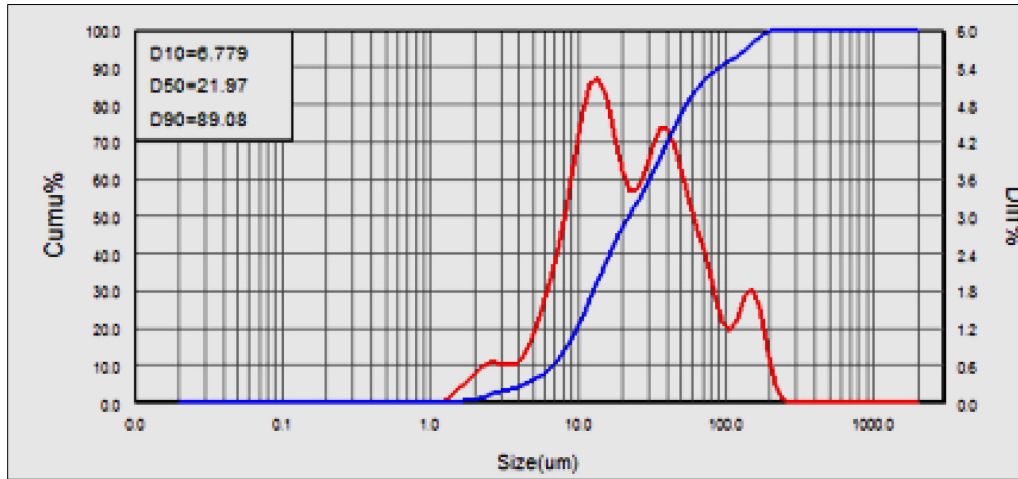
According to the above, it is clear that silica has been used as filler in epoxy composite, but limited studies have used multiple sources of silica as well as sources of silica resulting from the waste of other materials or by-products. Therefore, the present study aims to investigate the waste material (glass, fly ash) influences, including the high silica content and the filler addition upon the characteristics of the epoxy-based composites. Results of the epoxy with glass and fly ash addition will be compared with the specimens for the epoxy with silica fume addition.

Experimental Parts

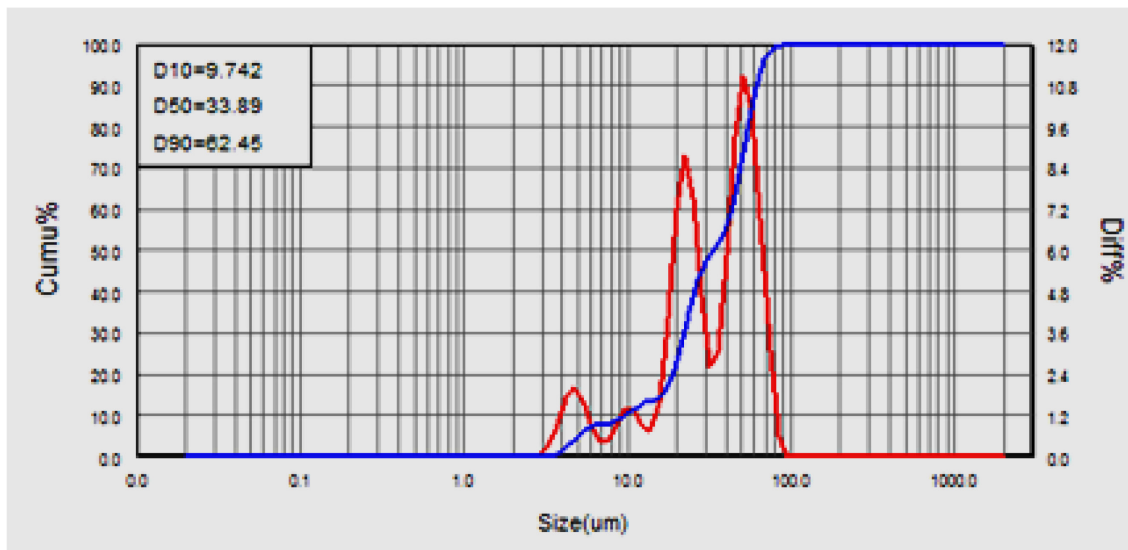
Commercially available epoxy resin (Sikadur-52, Sika Australia Pty Limited) along with hardener was utilized as a matrix material in the synthesis of various samples. Three types, including (silica fume, glass, and fly ash) powders were used as reinforcements. Broken glass and Fly ash were supplied from local sources. Glass was ground to a fine powder and then sieved. The silica fume (Fluka Company, Switzerland) used in this experiment contained (94.6%) SiO_2 . The analysis of the particle size of glass, silica fume and fly ash powder is depicted in Fig.1. The chemical analyses of Fly Ash and Glass Powder were done via an XRF microprobe analyzer, as displayed in Table 1.



a-



b-



c-

Fig. 1. The analysis of particle size for a- silica fume, b- fly ash, and c- glass powder

Table 1. The chemical analyses of Fly Ash and Glass Powder

	SiO ₂	MgO	K ₂ O	CaO	Al ₂ O ₃	Cl	Fe ₂ O ₃	Na ₂ O	SO ₃	CuO	P ₂ O ₅	ZnO
Fly ash	86.90	3.16	3.20	1.96	0.74	0.14	1.69	0.51	1.01	-	0.17	0.25
Glass	73.98	3.72	0.25	10.16	0.73	0.04	0.30	9.97	0.24	0.23	0.03	0.13

The filler material was gradually added to epoxy resin (part A) under vigorous stirring using a magnetic stirrer (SH-2 model) to produce a homogeneous mixture. After that, the hardener (part B) was added according to the mix ratio (2:1) provided by the adhesive manufacturer. Three groups of samples were prepared with (0, 5, 10, 15, and 20) wt% addition from fume silica, glass, and fly ash.

The chemical composition for the prepared samples (pure, 20% fume silica, 20% glass, 20% fly ash) was examined by Fourier transform infrared ((IRAffinity-1 device type).

For all epoxy samples, the micro-hardness was measured by a digital micro Vickers hardness tester (TH-717 model). The micro-hardness test was carried out for all prepared samples. The test was conducted by Digital Micro Vickers Hardness Tester (TH -717 model) with a square base diamond pyramid using a load of 0.24 N for 20 sec. The value of micro-hardness was taken as the average of (3) readings for every sample.

A wear test for a specimen of (15 mm) diameter and (6 mm) height was conducted for every sample of composite depending on the (ASTM (G99-04)) [28]. First, specimens were weighed by a

sensitive electric scale (M254A model) with (+0.0001) accuracy. The concept of Pin on Disk was utilized for studying dry wear using a wear tester device (type MT-4003, version 10.0). Wear test was performed at room temperature without using the lubricant. Dry sliding wear was studied by using the pin on disk concept with a normal force of (20 N) on the pin and a (300 rpm) disk rotating speed of disk. The sample was weighed after (5, 10, 15, and 20) min for determining the loss of weight. After that, the loss of weight was changed to the loss of volume using this equation:

$$\text{The loss of volume (mm}^3\text{)} = \text{The loss of weight (g)} / \text{Density (g/mm}^3\text{)}$$

Where:

$$\text{The loss of weight} = \text{The weight before the test} - \text{The weight after the test.}$$

Results and Discussion

The analysis of Fourier transform infrared was utilized for investigating the epoxy/micro powder composite structure, as revealed in Figure 2. In the Fourier-transform infrared spectroscopy (FTIR) analysis for neat epoxy, Fig. 2a, the characteristic peak at $\sim 3402\text{ cm}^{-1}$ is attributed to a hydroxyl group (O–H). The molecular structure is verified from the bonds of the reflection of contracting and stretching in the epoxy rings phase, noticed at around (1242 cm^{-1}) and (825 cm^{-1}) [6]. The peaks related to aromatic rings appeared at (1604) and (1512 cm^{-1}). The Aliphatic (C–H) stretching vibrations can be noted in the ($2800\text{--}3000\text{ cm}^{-1}$) range [6]. All these energy band results confirmed the structure of the neat epoxy.

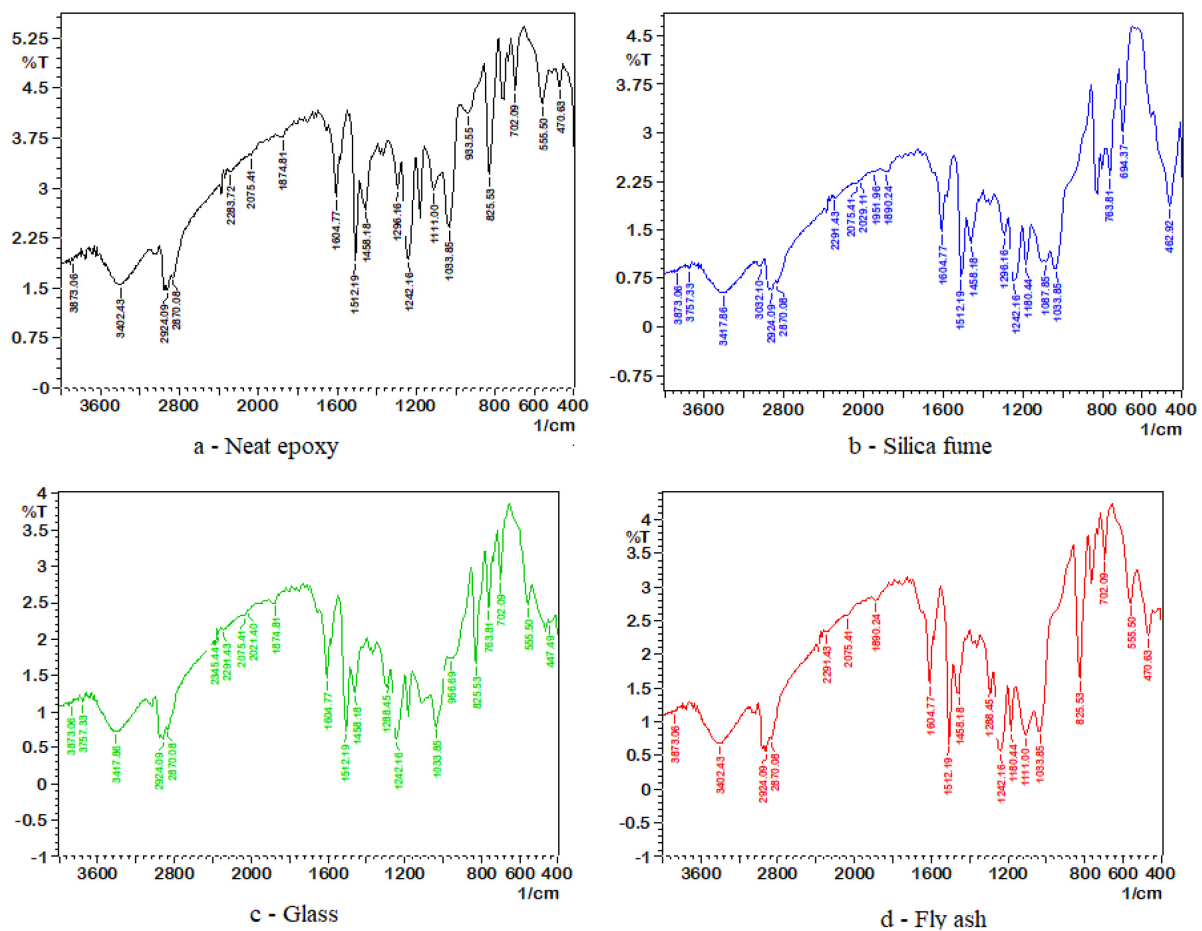


Fig. 2. FTIR analysis: a- neat epoxy, b- 20% silica, c- 20% glass, and d- 20% fly ash.

FTIR transmittance spectra of epoxy/micro powder composite with (20% silica fume, 20% glass, and 20% fly ash) weight ratio, shown in Fig. 2b,2c, and 2d, were achieved to investigate from the silica bands in epoxy. The analysis results manifested that the majority of the transmission of peaks can be traced back to the original epoxy. Also, it can be seen the appearance of the new band peaks of silica. Characteristics of the vigorous bands of transmission at (825 cm^{-1}) and (1087 cm^{-1}) are

ascribed to asymmetric, symmetric, and stretching vibration of Siloxane group (Si–O–Si) in the Silica, correspondingly, which verified the network of (SiO₂) [29]. Another new band peak at (3757 cm⁻¹) is attributed to the Si-O-H band in silica. The epoxide group in epoxy will react with the (SiO₂) Silanol group by H-bonding if micro silica is reacted with the epoxy. Where, it can be observed, from the neat epoxy resin spectra as well as its composites of silica fume, glass, and fly ash, that the OH groups stretching vibrations of epoxy resin was moved to (3417 cm⁻¹) for silica fume and glass-reinforced epoxy [29,30].

The other thing that is noticed from the figures is that the amount of transmission decreased by adding the silica fume powder and in a lesser percentage, by adding the glass and fly ash powder. The reduction in the transmission of radiation is due to the interaction of radiation with the powder and the occurrence of absorption and dispersion.

The hardness of the micro-powder-filled epoxy matrix composites has been studied. A load of 0.24 N for 20 msec was used to conduct the Vickers Hardness test on the samples. With the variation of filler content in the epoxy, the variation of hardness is shown in Fig.3. In most cases, the micro-hardness is improved by reinforcing the micro silica fume, glass, and fly ash in an epoxy polymer. Such improvement was owing to the vigorous H-bonding between the OH-groups in the parent matrix and Silanol (Si-OH) groups upon the surface of SiO₂, which limits the mobility of the chain and as a result improves the crosslinking [31]. It was observed that the values of hardness increased with increasing the micro-powder filler, this corresponds to the uniform dispersion of micro filler and the strong adhesion between micro powder and epoxy. Also, it can be noted that the hardness values of glass/epoxy composite were higher than those for the fly ash/epoxy and silica/epoxy composite, which is due to the hardness of the glass powder and also to the strength of the bond between the powder and the epoxy.

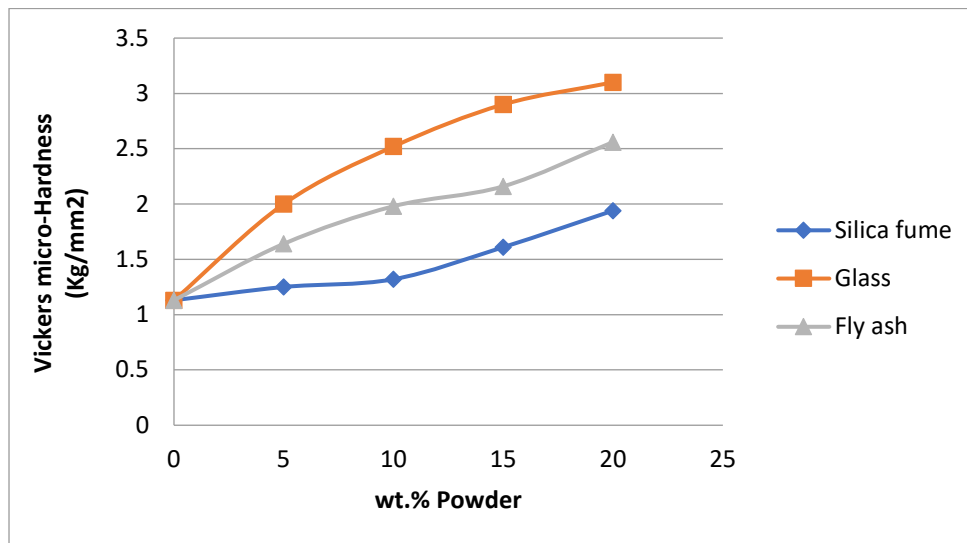
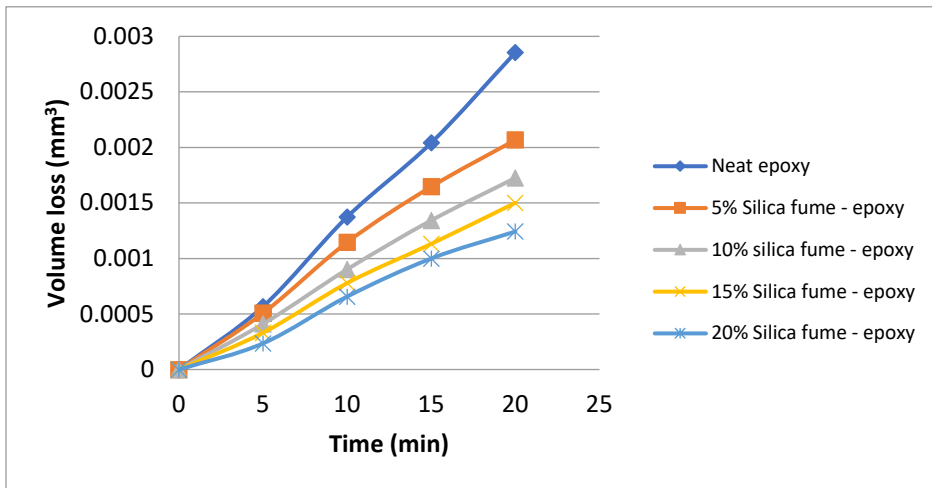


Fig.3. Vickers microhardness for all composite specimens.

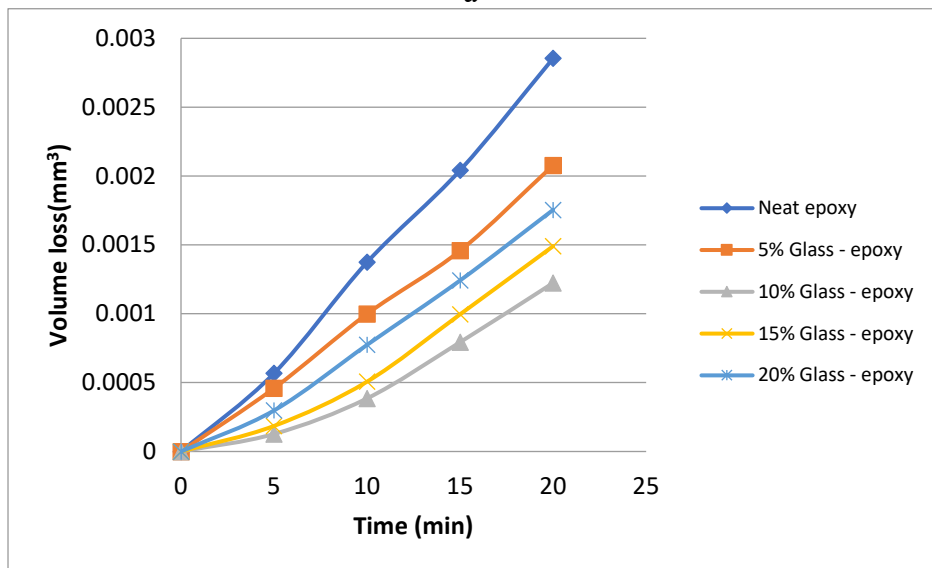
Converting the weight loss into a volume loss using every sample density was done. Fig. 4a to Fig.4c elucidate the test results at the similar circumstances stated before ($F = 20$ N), ($\omega = 200$ rpm) and ($t = 5, 10, 15,$ and 20 min). In such figures, it's obvious that the loss of volume is raised with the raising of the time of test, where the uppermost loss of volume was documented at (20) min. This is a predicted behavior, where the increase of the loss of particles of sample is with the raising of the time of friction between the sample surface and the rotating disk.

Further, these figures evince the effect of different particles addition on the wear rates at different materials. From Fig. 4a, for the silica–epoxy composite, it has been shown that there is a decrease in the wear rate of the composite samples with an increase in the silica particle additives. The increase in reinforcement has resulted in the increased hardness and caused the surface barrier for the penetration of hard asperities into the surface. Fig. 4b exhibits the effect of glass content on the wear rate of epoxy. One can notice that the loss of volume reduced radically with the rising percentage of

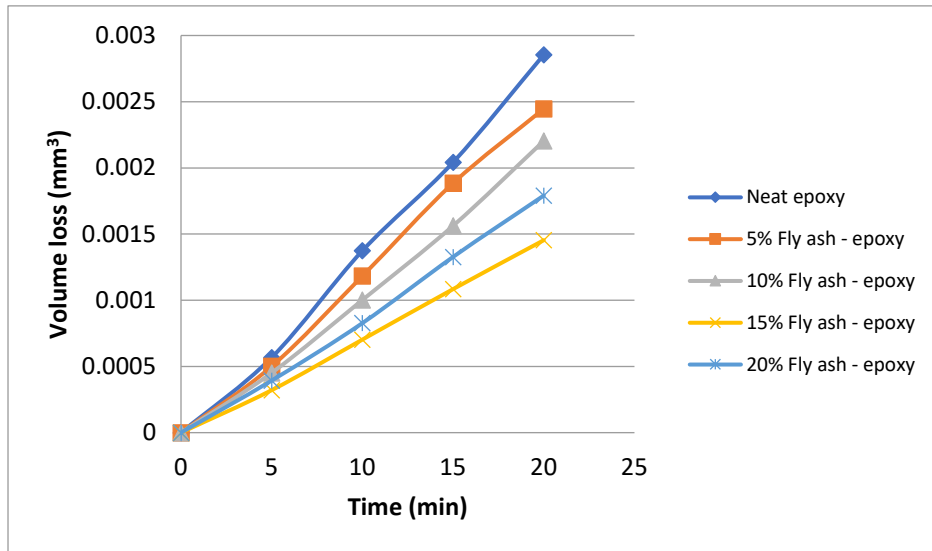
glass; even it attained to (10%) glass. That is perhaps owing to the glass particles' role in obstructing the motion of dislocation, thus, the wear resistance is also increased. It is noted that with an increase in the amount of added glass over 10%, the wear rate begins to increase, this is because increasing the amount of glass leads to an agglomeration of particles and thus to the heterogeneity of the composite material, which therefore affects the increase in roughness. The addition of fly ash and its effect on the wear resistance is illustrated in Fig.4c. The wear test results are close to the silica fume results, as the wear resistance increases with the addition of the fly ash particles. In fly ash – epoxy composite, the maximum wear rate is at 15% fly ash addition, and after this percentage, the wear rate begins to increase. The reason is due to the increase in the amount of fly ash, which leads to an agglomeration of particles and thus to the heterogeneity of the composite material.



a-



b-



c-

Fig. 4. Wear rate of composite specimens; a- Silica – epoxy composite, b- Glass – epoxy composite, and c- Fly ash – epoxy composite.

Fig. 5 demonstrates the wear rate (mm^3/min) in steady-state at 20 minute time. It is observed that there is converge in the results for wear rate in the case of silica fume, glass, and fly ash addition. The wear rate decreased with the increase of silica fume, glass, and fly ash addition. In the case of the fly ash addition, the minimum wear rate was at 15%, and after this percentage, the wear rate increased. However, in the case of glass addition, the minimum wear rate was at 10%, and after this percentage, the wear rate increased.

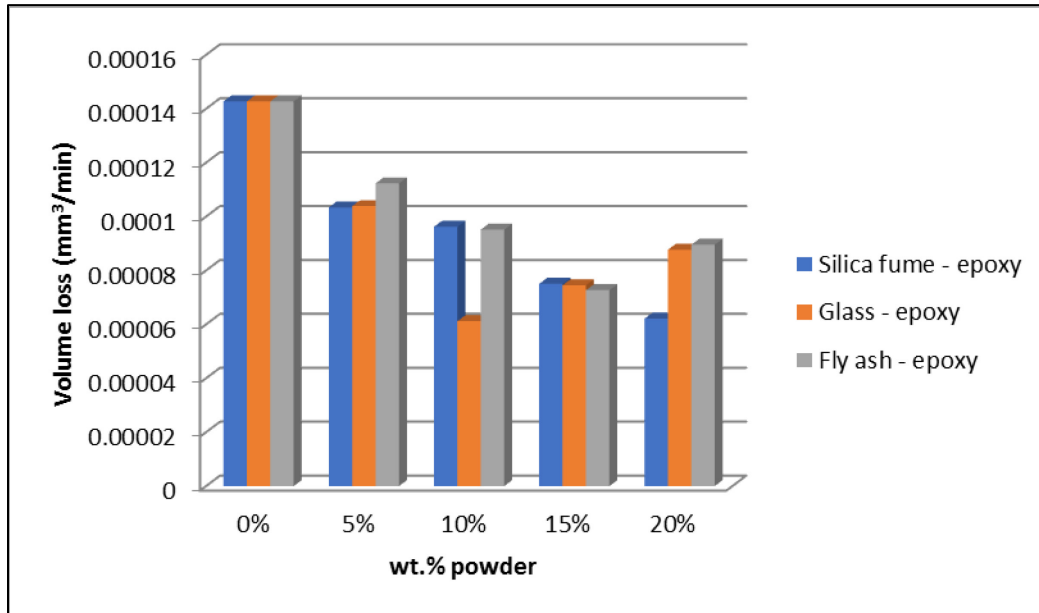


Fig. 5. Wear rate at steady state for the composite specimens at (time = 20 min).

Conclusions

Waste materials with high silica content were used as reinforcing filler in the epoxy matrix. The results for the glass/epoxy and fly ash/epoxy experiments were in good agreement with the results obtained in the silica fume/epoxy composite experiments. It was observed that the values of hardness increased with increasing the micro-powder filler, this corresponds to the uniform dispersion of micro filler and the strong adhesion between micro powder and epoxy. Also, it can be noted that the hardness values of glass/epoxy composite were higher than those for the fly ash/epoxy and silica/epoxy composite, which is due to the hardness of the glass powder and also to the strength of the bond between the powder and the epoxy. The wear rate decreased with the increase of the silica fume, glass, and fly ash addition. If fly ash was added, the minimum wear rate was at 15%, and after this percentage, the wear rate increased. However, if the glass was incorporated, the minimum wear rate was at 10%, and then was raised beyond that. In a conclusion, the best mixture that improved wear rate was when using 10% glass

References

- [1] A.H.R. Junior, J.E. Gomez, D.W. Hale, M.L.P. Tonatto, T.H. Panzera, Effects of silica microparticles in glass fiber/epoxy laminates, in 4th Brazilian Conf. Compos. Mater., Rio de Janeiro, 2018.
- [2] A.S. Mostovoy, A. V Yakovlev, Reinforcement of epoxy composites with graphite-graphene structures, *Sci. Rep.* 9 (2019) 1–9.
- [3] C. Jayaseelan, P. Padmanabhan, A. Athijayamani, K. Ramanathan, Comparative investigation of mechanical properties of epoxy composites reinforced with short fibers, macroparticles, and microparticles, *BioResources* 12 (2017) 2864–2871.
- [4] P. Deogonda, V.N. Chalwa, Mechanical property of glass fiber reinforced epoxy composites, *Int. J. Sci. Eng. Res.* 1 (2013) 2347–3878.
- [5] T. Uygunoglu, I. Gunes, W. Brostow, Physical and mechanical properties of polymer composites with a high content of wastes including boron, *Mater. Res.* 18 (2015) 1188–1196.
- [6] F. Ali, N. Ali, M. Altaf, A. Said, S.S. Shah, M. Bilal, Epoxy polyamide composites reinforced with silica nanorods: fabrication, thermal and morphological investigations, *J. Inorg. Organomet. Polym. Mater.* 30 (2020) 3869–3877.
- [7] L.M. Johnson, E. Ledet, N.D. Huffman, S.L. Swarner, S.D. Shepherd, P.G. Durham, G.D. Rothrock, Controlled degradation of disulfide-based epoxy thermosets for extreme environments, *Polymer (Guildf)*. 64 (2015) 84–92.
- [8] D. Zhang, J. Wang, S. Wen, P. Wang, C. Yin, Z. Du, Preparation of Silica Powder in Epoxy Resin Wear-Resistant Coating, *Adv. Mater. Phys. Chem.* 5 (2015) 60.
- [9] C.N.A. Jaafar, I. Zainol, O.O. Aremu, Effect of silica fillers on mechanical properties of epoxy/kenaf composites, in *J. Phys. Conf. Ser.*, IOP Publishing, 2018: p. 12006.
- [10] D. Tzetzis, K. Tsongas, G. Mansour, Determination of the mechanical properties of epoxy silica nanocomposites through FEA-supported evaluation of ball indentation test results, *Mater. Res.* 20 (2017) 1571–1578.
- [11] Y. Sekiguchi, Effects of mixed micro and nano-silica particles on the dynamic compressive performances of epoxy adhesive, *Appl. Adhes. Sci.* 5 (2017) 1–12.
- [12] J.-J. Park, Effect of silica particle size on the mechanical properties in an epoxy/silica composite for HV insulation, *Trans. Electr. Electron. Mater.* 13 (2012) 248–251.

- [13] C.M. Manjunatha, N. Jagannathan, K. Padmalatha, A.C. Taylor, A.J. Kinloch, The effect of micron-rubber and nano-silica particles on the fatigue crack growth behavior of an epoxy polymer, *Int. J. Nanosci.* 10 (2011) 1095–1099.
- [14] Z.F. Jawad, R.J. Ghayyib, A.J. Salman, Microstructural and Compressive Strength Analysis for Cement Mortar with Industrial Waste Materials, *Civ. Eng. J.* 6 (2020) 1007–1016.
- [15] N. Permatasari, T.N. Sucahya, A.B.D. Nandiyanto, Agricultural wastes as a source of silica material, *Indones. J. Sci. Technol.* 1 (2016) 82–106.
- [16] A.A. Shubbar, M. Sadique, M.S. Nasr, Z.S. Al-Khafaji, K.S. Hashim, The impact of grinding time on properties of cement mortar incorporated high volume waste paper sludge ash, *Karbala Int. J. Mod. Sci.* 6 (2020) 396–403.
- [17] M.S. Nasr, I.M. Ali, A.M. Hussein, A.A. Shubbar, Q.T. Kareem, A.T. AbdulAmeer, Utilization of locally produced waste in the production of sustainable mortar, *Case Stud. Constr. Mater.* 13 (2020) e00464.
- [18] M. Abdulredha, A. Abdulridha, A.A. Shubbar, R. Alkhaddar, P. Kot, D. Jordan, Estimating municipal solid waste generation from service processions during the Ashura religious event, in *IOP Conf. Ser. Mater. Sci. Eng.*, IOP Publishing, 2020: p. 12075.
- [19] H.S. Majdi, A.A. Shubbar, M.S. Nasr, Z.S. Al-Khafaji, H. Jafer, M. Abdulredha, Z. Al Masoodi, M. Sadique, K. Hashim, Experimental data on compressive strength and ultrasonic pulse velocity properties of sustainable mortar made with high content of GGBFS and CKD combinations, *Data Br.* 31 (2020) 105961.
- [20] M.S. Nasr, A.A. Shubbar, Z.A.-A.R. Abed, M.S. Ibrahim, Properties of eco-friendly cement mortar contained recycled materials from different sources, *J. Build. Eng.* 31 (2020) 101444.
- [21] A.A. Shubbar, H. Jafer, M. Abdulredha, Z.S. Al-Khafaji, M.S. Nasr, Z. Al Masoodi, M. Sadique, Properties of cement mortar incorporated high volume fraction of GGBFS and CKD from 1 day to 550 days, *J. Build. Eng.* 30 (2020) 101327.
- [22] M.S. Nasr, T.H. Hussain, H.Z. Kubba, A.A. Shubbar, Influence of using high volume fraction of silica fume on mechanical and durability properties of cement mortar, *J. Eng. Sci. Technol.* 15 (2020) 2492–2506.
- [23] M.K. Obaid, M.S. Nasr, I.M. Ali, A.A. Shubbar, K.S. Hashim, Performance of Green Mortar Made from Locally Available Waste Tiles and Silica Fume, *J. Eng. Sci. Technol. Technol.* 16 (2021).
- [24] M.S. Nasr, Z.A. Hasan, M.K. Abed, M.K. Dhahir, W.N. Najim, A.A. Shubbar, H.Z. Dhahir, Utilization of High Volume Fraction of Binary Combinations of Supplementary Cementitious Materials in the Production of Reactive Powder Concrete, *Period. Polytech. Civ. Eng.* 65 (2021) 335–343.
- [25] Z.A. Hasan, M.S. Nasr, M.K. Abed, Properties of reactive powder concrete containing different combinations of fly ash and metakaolin, *Mater. Today Proc.* (2021).
- [26] A.A. Shubbar, D. Al-Jumeily, A.J. Aljaaf, M. Alyafei, M. Sadique, J. Mustafina, Investigating the Mechanical and Durability Performance of Cement Mortar Incorporated Modified Fly Ash and Ground Granulated Blast Furnace Slag as Cement Replacement Materials, in *2019 12th Int. Conf. Dev. systems Eng.*, IEEE, 2019: pp. 434–439.
- [27] Z.A. Hasan, M.K. Abed, M.S. Nasr, Studying the Mechanical Properties of Mortar Containing Different Waste Materials as a Partial Replacement for Aggregate, *Int. Rev. Civ. Eng.* 10 (2019) 155–161.

[28] ASTM G99-05, Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus, ASTM International, West Conshohocken, PA, 2005.

[29] I.M. Joni, L. Nulhakim, M. Vanitha, C. Panatarani, Characteristics of crystalline silica (SiO₂) particles prepared by simple solution method using sodium silicate (Na₂SiO₃) precursor, in: J. Phys. Conf. Ser., IOP Publishing, 2018: p. 12006.

[30] S. Soontaranon, W. Limphirat, S. Pratapa, XRD, WAXS, FTIR, and XANES studies of silica-zirconia systems, *Ceram. Int.* 45 (2019) 15660–15670.

[31] H. Khan, M. Amin, M. Ali, M. Iqbal, M. Yasin, Effect of micro/nano-SiO₂ on mechanical, thermal, and electrical properties of silicone rubber, epoxy, and EPDM composites for outdoor electrical insulations, *Turkish J. Electr. Eng. Comput. Sci.* 25 (2017) 1426–1435.