



Surface Morphology Analysis using Atomic Force Microscopy and Statistical Method for Glass Fiber Reinforced Epoxy-Zinc Oxide Nanocomposites

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Abstract

Nanoparticle-filled fiber-reinforced polymer composites are gaining widespread application due to the exhibition of peculiar and unique properties that can be tailored to suit specific requirements. The current paper discusses the fabrication of glass fiber-reinforced epoxy-zinc oxide (EGZ) nanocomposites. The inclusion of hard ceramic zinc oxide (ZnO) nanoparticles is known to improve the mechanical, thermal, and optical properties of the composites but also tends to increase the surface roughness. Evaluating surface roughness becomes important, especially if such material is used for applications that require smooth surfaces. Thus, an effort is made to study the surface roughness of the fabricated EGZ nanocomposite using atomic force microscopy (AFM). The ZnO nanoparticles are ultrasonically blended with epoxy resin in 1, 2, and 3% weight fractions. Compression molding is used to create the nanocomposite laminates. The predictors include sonication time, compression time, and ZnO nanofiller content. The influence of nanofiller content on the amplitude functional parameter is also statistically investigated. High-fit and prediction-ability linear regression models are designed and verified for predicting surface roughness characteristics within the experimental restrictions. All amplitude functional parameters of surface roughness were higher in nanocomposites having higher ZnO content.

Keywords: Zinc oxide; Polymer nanocomposites; Surface roughness; Atomic force microscopy; Analysis of variance.

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1. Introduction

Combining two or more raw materials to make a unique composition that can be used as an intermediate or a final product has been around for a long time and has a wide range of applications.^[1] However, in order to make a formulation that works well and meets a user's needs, it is important to know about the material properties and the processing parameters that must be used when mixing and/or combining different ingredients that are either naturally occurring or made synthetically, and are often incompatible with each other.^[2,3] The final product comprises different phases that cannot be mixed. Such formulations appear to be homogeneous macroscopically, but they are very different microscopically. In another type of formulation, two or more components are mixed to make a new material with better qualities than the parts that make up the composite. Polymer composites,

because they can be made to have different strengths and weights, are now used in a wide range of applications, from earth to space. Polymer composites can be made by adding different types of reinforcements. This is a good way to get around the limitations of the polymer material.^[4-6] Reinforcements in the form of continuous fibers are thought to be a good way to transfer the loads and stresses from the weaker polymer matrix to the stiffer fibers. Glass fibers are used a lot to make polymer composites because they have good mechanical properties at a good price.^[7] It has become more common for nanotechnology to be used in materials science as it has opened up a wide range of high-performance materials.^[8-13] Polymer nanocomposites are a new engineering and functional category of materials made by adding nanoparticles as fillers to different types of polymer matrices. However, it is important to note that they can achieve these superior properties only if the materials are processed with extreme care and control. Also, the final material properties of the polymer nanocomposite largely depend on how well the chosen nanofillers are spread out in the matrix.^[14] Ultrasonication is the best way to ensure the nanofillers are

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evenly distributed in the matrix.^[15-17] The ultrasonication technique begins with the addition of nanoparticles to the polymeric resin, followed by the vigorous stirring of the solution using an ultrasonic-vibrating sonicator probe. The critical parameter that can affect the final material property in this process is the sonication time. This is because when the sonication period increases, the resin's temperature and viscosity tend to increase as well.^[18] These circumstances may present difficulties during composite production, particularly if wet processing is used. As a result, it is critical to carefully set the sonication period to guarantee that the changed resin can be handled easily during the fabrication process. A significant disadvantage of polymer matrix composites is their low heat stability. Thus, it is critical to understand the glass transition temperature of the polymeric resin since it indicates the point at which the polymeric matrix loses its stiffness and becomes soft/rubbery. In the case of thermosets, the resin becomes hard only after the curing process is complete due to the formation of strong covalent connections.^[19] Thus, in the case of the thermoset matrix, the curing period is a critical factor in determining the final composite characteristics. Hard ceramic nanoparticles are added as fillers to compensate for the polymeric resins' low-temperature stability. While these inclusions are advantageous in some ways, they often result in a rough and hard surface on the composite materials. The roughness of the surface becomes a key factor in a variety of technical applications and must therefore be properly controlled and maintained within specified limits. In this study, hard ceramic ZnO nanofiller is added to the epoxy resin in different amounts to make glass fiber-reinforced epoxy-zinc oxide nanocomposites. The atomic force microscopy (AFM) analysis is used to figure out the different features of the surface topography. Also, analysis of variance (ANOVA) and

regression analysis is conducted to see if there is a difference in the average values and to create mathematical models that can predict surface roughness parameters. Fig. 1 represents the present study pictographically and has been explained in detail in the further sections of this article.

2. Materials and method

2.1 Materials

The matrix material used is a colorless, bisphenol-A type unmodified epoxy resin with a medium viscosity commercially known as Araldite LY556. A suitable curing agent known as Aradur HY951 is used to cure the resin. The unidirectional, woven roving mats of E-glass fibers are used as reinforcement. ZnO nanoparticles with an average particle size of 40 nm are utilized as nanofillers.

2.2 Fabrication of EGZ nanocomposites

Six square E-glass mats with a length of 300 mm and a width of 300 mm are cut from the roving in the first phase to create a 3 mm thick composite laminate. These square cut-out mats are weighed using an electronic balance. The obtained fiber weight is maintained consistently across all the fabricated composite laminates. ZnO nanoparticles are introduced into the resin at varying concentrations ranging from 1% to 3%. The weight fraction of reinforcement and matrix/matrix-filler is maintained as 50/50. As a result, the resin and filler weights are altered as specified in Table 1. As specified by the vendor, the resin-to-curing agent ratio should be maintained at 10:1. The appropriate amount of resin is placed in a glass beaker, and the required amount of ZnO nanoparticles is added at the specified weight percent. The ultrasonication process is used to blend the nanofillers into the epoxy resin,^[20] in which the solution of resin and filler is stirred using ultrasonic sound

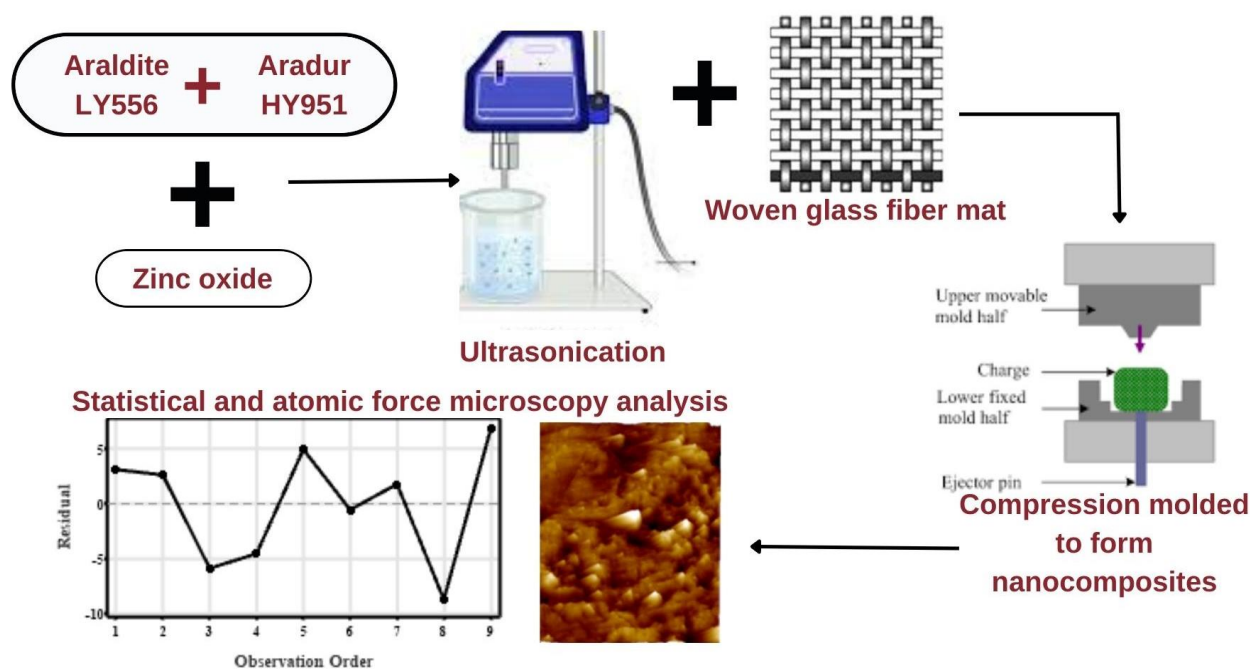


Fig. 1 Schematic representation detailing the present study.

waves at a frequency of 20 kHz delivered via a probe. Following sonication, the needed amount of curing agent is added to the resin/filler solution and composite laminates. ZnO nanoparticles are introduced into the resin at varying concentrations ranging from 1% to 3%. The weight fraction of reinforcement and matrix/matrix-filler is maintained as 50/50. As a result, the resin and filler weights are altered as specified in Table 1. As specified by the vendor, the resin-to-curing agent ratio should be maintained at 10:1. The appropriate amount of resin is placed in a glass beaker, and the required amount of ZnO nanoparticles is added at the specified weight percent. The ultrasonication process is used to blend the nanofillers into the epoxy resin,^[20] in which the solution of resin and filler is stirred using ultrasonic sound waves at a frequency of 20 kHz delivered via a probe. Following sonication, the needed amount of curing agent is added to the resin/filler solution and manually mixed using a wooden spatula.

The first square glass fiber mat is placed on a 1 mm thick Teflon sheet, and the resin/filler/curing agent solution is dispersed with a brush. The second square fiber mat is positioned on top of the first. As with the first mat, the resin/filler/curing agent solution is evenly spread throughout the second mat. This is followed by using a hand roller to remove any excess resin solution between the two mats gently. Similarly, all remaining mats with a uniform layer of resin/filler/curing agent solution are stacked one over the other with 0° fiber orientation. On top of the previous stacked mat, another Teflon sheet with a thickness of 1 mm is applied. The stacked laminates are compression molded at 100 °C for varying lengths of time.

The laminate and the Teflon sheets are placed on the compression molding machine's mold die and squeezed at 20 bar pressure. The laminates are removed from the compression molding machine and allowed to cure at room temperature fully. Table 1 shows how the various composite laminates manufactured in this work are coded for ease of identification.

Table 1. Composite sample coding concerning the composition of glass fiber-reinforced epoxy-zinc oxide (EGZ) nanocomposites.

Composite coding	Glass fibers (g)	Epoxy LY556 (g)	ZnO Nanofiller (g)
EGZ1	253	250.47	2.53
EGZ2	253	247.94	5.06
EGZ3	253	245.41	7.59

2.3 Experimental details

It is determined that the polymer-filler matrix has a significant effect on the roughness properties of the polymer nanocomposite materials developed with them.^[21] There are numerous ways of nano emulsification available to produce polymer-filler matrixes, which are broadly classified as high and low-energy methods. Ultrasonication, amongst them – a high-energy approach, is widely applied due to its operational

advantages.^[22] Moreover, it is an efficient technique for creating nanoemulsions that allow precise control of the emulsion properties. It can be used to create a nanoemulsion *in situ* or to minimize the size of an emulsion that has already been prepared.^[23] The present work analyzes two critical parameters involved in the preparation of EGZ nanocomposites: sonication time, which is involved in the formation of the polymer-filler matrix, and compression time, which is involved in the overall formation of the EGZ laminate and evaluates their effect on the surface roughness of the EGZ nanocomposites created using the developed polymer-filler matrix. Three different fixed conditions are considered as given in Table 2. All the composite sample types listed in Table 1 are subjected to all the conditions listed in Table 2.

Table 2. Fixed conditions of predictor variables for experimental purposes.

Condition Number	Sonication time (minutes)	Compression time (minutes)
1	10	5
2	15	10
3	20	15

2.4 Surface roughness evaluation

Surface roughness assessment is useful for several basic material properties such as friction, contact deformation, heat and electric current conduction, contact joint tightness, and positional accuracy. As a result, surface roughness evaluation has been the focus of theoretical and empirical studies for many years. The geometry of the real surface is sufficiently complex that a finite set of parameters cannot offer a complete description. A more precise description can be obtained by increasing the number of parameters used for its evaluation. Surface roughness parameters are often classified into three types based on their functionality. These are divided into three categories: amplitude parameters, spacing parameters, and hybrid parameters.^[24] Thus, the current work focuses on determining surface roughness using three amplitude parameters: arithmetic average height (R_a), root mean square roughness (R_q), and profile maximum height (R_t). Atomic force microscopy (AFM) is a well-known technique for analyzing the surfaces of micro/nanostructured coatings.^[25] AFM is an effective technique for characterizing nanoparticles and nanomaterials because it offers qualitative and quantitative information on several physical properties such as size, shape, surface texture, and roughness.^[26] Additionally, it is a technology capable of imaging nearly any surface type, including polymers, ceramics, composites, glass, and biological materials.^[27]

Thus, the surface topographical studies are carried out in the present study through atomic force microscopy (AFM) to determine the impact of nanofillers on the surface characteristics of the fabricated EGZ nanocomposites. The AFM images of the EGZ nanocomposites are obtained, and the surface roughness amplitude functional parameters R_a , R_q ,

and R_t are measured using Equations 1, 2, and 3, respectively.

$$R_a = \frac{1}{L} \int_0^L |Z(x)| dx \tag{1}$$

$$R_q = \sqrt{\frac{1}{L} \int_0^L |Z^2(x)| dx} \tag{2}$$

$$R_t = \frac{\sum_1^5 (R_p) + \sum_1^5 R_v}{5} \tag{3}$$

2.5 ANOVA analysis and regression modeling

Analysis of variance (ANOVA) is a conceptually straightforward, effective, and widely used technique for performing statistical testing on studies involving two or more groups.^[28] Since the present work comprises more than two groups^[29] in terms of conditions mentioned earlier in Table 2, the ANOVA test is used to investigate the significant differences among the group averages.^[30] Regression analysis is a popular statistical learning technique used for determining the relationship between a dependent variable Y and p independent variables $X = [X_1, \dots, X_p]$. The variables X_k ($k = 1, \dots, p$) are known as predictors, explanatory variables, or covariates, while the dependent variable Y is also known as the response variable or outcome.^[31] In a nutshell, it is the

study of how one or more predictors influence a response variable.^[32] In the present work, the ‘conditions’ as mentioned earlier comprising the combination of sonication time and compression time is considered as a categorical pooled factor, and filler weight percentage is taken as the continuous predictor. Using ANOVA, the significance of filler material addition and the condition is checked for the amplitude functional factors of surface roughness. Based on the significance and contribution of the selected factors, regression analysis is further employed to develop the predicting mathematical model. The model is later validated using the additional set of experiments.

3. Results and discussion

3.1 AFM results

Figure 2 shows three-dimensional AFM images of EGZ nanocomposites. Equations 1, 2, and 3 are used to calculate the surface roughness parameters of the fabricated EGZ nanocomposites. The needed amplitude functional surface roughness values for manufactured EGZ nanocomposites are listed in Table 3 for varied sonication and compression times.

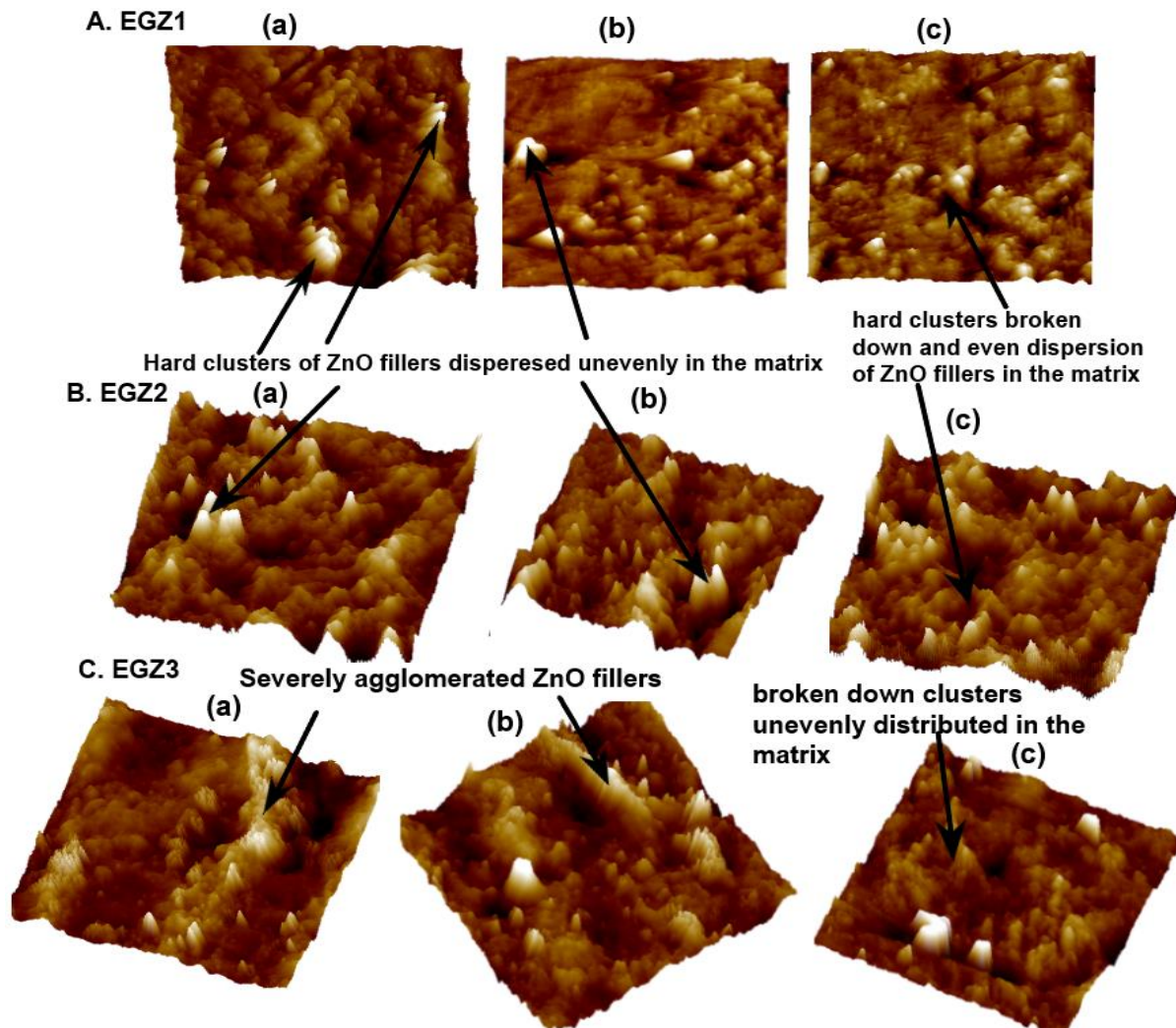


Fig. 2 AFM 3D scans of EGZ nanocomposites: (a) condition 1 – sonication time of 10 min and compression time of 5 min; (b) condition 2 – sonication time of 15 min and compression time of 10 min; (c) condition 3 – sonication time of 20 min and compression time of 15 min.

Table 3. Amplitude functional parameters of surface roughness measured for EGZ nanocomposites.

ZnO Weight% in the polymer-filler matrix	Sonication time St (min)	Compression time Ct (min)	Arithmetic average roughness, Ra (μm)	Root mean square, Rq (μm)	Maximum height of the profile, Rt (μm)
1	10	5	47.4	67.1	874
2	10	5	92.9	126	1517
3	10	5	119	134	1726
1	15	10	49	72	900
2	15	10	98	135	1573
3	15	10	123	153	1811
1	20	15	45	82	822
2	20	15	90	150	1445
3	20	15	100	116	1500

3.2 Results of ANOVA and regression analysis

The analysis of variance results for the arithmetic average roughness, root mean square, and maximum profile height of EGZ nanocomposites under all three conditions are shown in Tables 4, 5, and 6. This analysis was conducted at a 5% level of significance, which corresponds to a 95% confidence level. The fourth column of the tables provides each factor's percentage contribution (P) to the overall variation, showing its degree of influence on the outcome.

3.3 Mathematical modeling for R_a , R_q , and R_t

Equations 4, 5, and 6 in Table 7 provide the mathematical relationship between the arithmetic average roughness and the nanofiller weight percentage utilized in the polymer-filler matrix.

Equations 7, 8, and 9 in Table 8 provide the mathematical relationship between the root mean square and the nanofiller weight percentage utilized in the polymer-filler matrix:

Equations 10, 11, and 12 in Table 9 provide the mathematical relationship between the maximum profile height and the nanofiller weight percentage utilized in the polymer-filler matrix:

3.4 Analysis of AFM results

3.4.1 Effect of filler content sonicated to the matrix

The AFM analysis results in Table 2 indicate that the amplitude functional parameters of surface roughness are influenced by the nanofiller (ZnO) loading and that these parameters increase as the ZnO weight percent in the epoxy matrix increases. The 3D AFM scans in Fig. 1 demonstrate

Table 4. ANOVA results for arithmetic average roughness of EGZ nanocomposites using AFM analysis.

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Regression	3	6485.27	99.90%	6485.27	2161.76	1626.20	0.000
F%	1	6246.83	96.22%	6246.83	6246.83	4699.22	0.000
Condition	2	238.44	3.67%	238.44	119.22	89.68	0.000
Error	5	6.65	0.10%	6.65	1.33		
Total	8	6491.92	100.00%				

Table 5. ANOVA results for root mean square of EGZ nanocomposites using AFM analysis.

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Regression	3	9715.00	99.99%	9715.00	3238.33	26763.10	0.000
F%	1	8809.00	90.67%	8809.00	8809.00	72801.67	0.000
Condition	2	906.00	9.33%	906.00	453.00	3743.81	0.000
Error	5	0.61	0.01%	0.61	0.12		
Total	8	9715.61	100.00%				

Table 6. ANOVA results for maximum profile height of EGZ nanocomposites using AFM analysis.

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Regression	3	953522	99.98%	953522	317841	7163.95	0.000
F%	1	929053	97.41%	929053	929053	20940.35	0.000
Condition	2	24469	2.57%	24469	12234	275.76	0.000
Error	5	222	0.02%	222	44		
Total	8	953744	100.00%				

Table 7. The mathematical model for determining the arithmetic average roughness of EGZ nanocomposites for a given filler weight content at varying sonication and compression time.

Condition	Mathematical Model	Equation Number
1 – Sonication time – 10 min; Compression time – 5 min	$R_a = 10.47 + 32.267F\%$	(4)
2 – Sonication time – 15 min; Compression time – 10 min	$R_a = 18.90 + 32.267F\%$	(5)
3 – Sonication time – 20 min; Compression time – 15 min	$R_a = 22.80 + 32.267F\%$	(6)

Table 8. The mathematical model for determining root mean square value of EGZ nanocomposites for a given filler weight content at varying sonication and compression time.

Condition	Mathematical Model	Equation Number
1 – Sonication time – 10 min; Compression time – 5 min	$R_q = 29.067 + 38.317F\%$	(7)
2 – Sonication time – 15 min; Compression time – 10 min	$R_q = 38.033 + 38.317F\%$	(8)
3 – Sonication time – 20 min; Compression time – 15 min	$R_q = 53.367 + 38.317F\%$	(9)

Table 9. The mathematical model for determining the maximum profile height of EGZ nanocomposites for a given filler weight content at varying sonication and compression time.

Condition	Mathematical Model	Equation Number
1 – Sonication time – 10 min; Compression time – 5 min	$R_t = 425.33 + 393.50F\%$	(10)
2 – Sonication time – 15 min; Compression time – 10 min	$R_t = 431.00 + 393.50F\%$	(11)
3 – Sonication time – 20 min; Compression time – 15 min	$R_t = 538.67 + 393.50F\%$	(12)

that with ZnO nanofiller loadings of 1% and 2%, the fillers are more distributed evenly inside the matrix with less clumping. However, when the nanofiller loading is increased to 3%, the nanofillers agglomerate significantly, and there is no uniform dispersion of the nanofiller within the matrix, as illustrated in Fig. 2(c). This can be linked to the nanofiller's cluster growth mechanism within the matrix.^[33] Nanoparticles cluster or aggregate into an agglomeration due to their large surface area and strong physical forces of attraction.^[34] The formation of agglomeration inside the matrix reduces the number of strong interfacial areas, which has a significant impact on the physical properties of polymer composites.^[35] As illustrated in Fig. 2(a), there is less possibility of agglomeration at a 1 wt.% loading of the ZnO nanofiller. In this case, the particles are few, and they have sufficient area to scatter within the matrix. Due to the small number of particles spread far apart, the physical force of attraction between them tends to be negated due to the increased particle-to-particle distance. When the ZnO nanofiller loading is increased to 2%, the particle-to-particle distance tends to decrease, and the particles cannot overcome the strong physical force of attraction. As a result of this scenario, the particles congregate and form a cluster, as illustrated in Fig. 2(b). However, at a loading of 3 wt.% ZnO nanofiller, substantial agglomeration of the particles occurs, as illustrated in Fig. 2(c), along with increased unevenness in cluster dispersion within the matrix. As the nanofiller loading percentage grows from 2 to 3 wt.%, not only particle-to-particle interaction occurs, but also cluster-to-cluster interaction. This occurrence leads to the formation of a bigger cluster of nanofiller that tends to concentrate in one area of the matrix, leaving the remainder of the matrix empty of filler and/or cluster of nanofiller.

3.4.2 Effect of sonication time

According to the surface roughness characteristics provided in Table 3, sonication time is an essential parameter that influences the dispersion of the ZnO nanoparticles inside the matrix and hence changes the surface topography of the glass fiber-reinforced epoxy-zinc oxide (EGZ) nanocomposites, as illustrated in Fig. 2.

During sonication, implosions induce higher temperatures and pressures extending to 5000 K and 1000 atm respectively in a time span of around 50 ns,^[36] causing shock waves to propagate through the resin/filler solution. By inducing eddy currents and severe shear stresses, these shock waves tend to dissolve surrounding filler particles, resulting in the creation of intense turbulence.^[37] When the clusters of ZnO nanoparticles break down, they are evenly spread out in the matrix, inferring that there are fewer loose hard ZnO nanoparticles that are found on the surface in this case. Thus, the roughness of the surface tends to go down as the sonication time goes up. In Fig. 2(a), for the sonication time of 10 min, the surface of the EGZ composites is made up of hard clusters of ZnO fillers that are spread out all over the matrix.

Sonication time increases to 15 min, and these hard clusters are broken down even more, with a little better dispersion than in the first case, as shown in Fig. 2(b). However, when the sonication time is increased to 20 min, the ZnO fillers are spread out more evenly, and there are fewer clumps, evidently depicted in Fig. 2(c).

3.4.3 Effect of compression time

Compression time is also important for high-quality composite laminates with better physical properties.^[38] Compression time is important because it can lead to flaws in the composite laminate and make the composite less durable.^[39] From Fig. 2, the surface roughness tends to go down as the compression time goes up. Since longer pressure is applied, the clusters of

nanofiller, if any, are broken down, and the resin that flows because of compression pressure picks up the broken-down particles and moves them along with the flow. This ensures that the nanofiller is spread out evenly and the surface roughness goes down. It also gets easier for micro-voids to get out of the interphases as the compression time goes up [40,41] and results in the formation of good quality composite laminate.

Figure 2(c) shows that the surface of the EGZ composites has more evenly distributed ZnO nanofillers. There are very few clusters of nanofillers on the surface of EGZ composites.

3.5 Analysis from ANOVA and regression analysis

It can be seen from ANOVA results presented in Table 4, Table 5, and Table 6 that filler weight percentage is significant for all the amplitude functional parameters of surface roughness. The sonication time and compression time pooled together as conditions; a categorical factor is also found to be significant in all the cases. As a result, mathematical prediction models developed for all amplitude functional parameters associated with all three circumstances are valid. R^2 , R^2 -adjusted, and R^2 -predicted values for all established equations are greater than 95%, indicating that the developed linear model possesses a high degree of fit and does not require additional predictors. Additionally, the model demonstrates a high degree of prediction accuracy.

The linear regression equation demonstrates that increasing the ZnO nanofiller content in the polymer-filler matrix by one weight percent increases the arithmetic average

roughness value by $32.267 \mu\text{m}$, the root mean square increases by $38.317 \mu\text{m}$, and the maximum profile height increases by $393.50 \mu\text{m}$, regardless of the conditional effect. Moreover, the conditions substantially affect the amplitude functional parameters of surface roughness, as indicated by the increasing trend of the intercept values in all regression models from conditions 1 to 3, as illustrated in Equations 4-12. Thus, statistical analysis confirms the AFM study and establishes that increasing the sonication duration, compression time, and filler weight percentage results in an increase in the surface roughness of the nanocomposites.

A Pareto chart is a type of column chart that is used to prioritize problem-solving steps to find which element has the greatest impact on the response variable.[42] Fig. 3 represents the Pareto chart of standardized effects for the measured values of surface roughness elements. From the Figs. 3(a), 3(b), and 3(c) represent the chart for arithmetic average roughness, root means square, and maximum profile height respectively, the factor A: filler content proves to have maximum effect. The residual plots for arithmetic average roughness, root mean square and maximum profile height are illustrated by Figs. 4, 5, and 6 respectively. The result indicating a highly significant effect of ZnO filler content on surface roughness morphology agrees with one of the prior studies by Ramezanzadeh *et al.*[43]

From the obtained residual plots for all three characterizing elements of surface roughness, it is seen that the variation between experimental and predicted values is very small, on the standard residual scale. Moreover, the frequency plots of

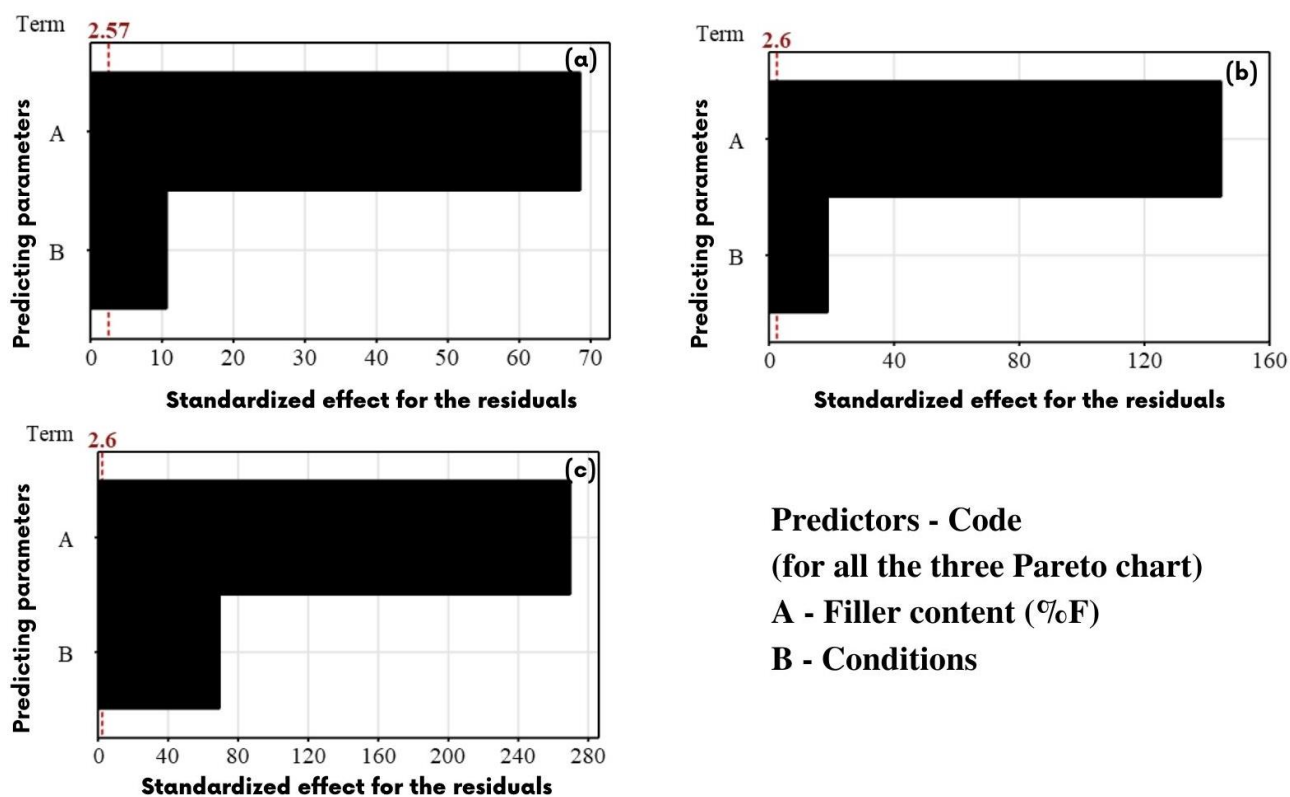


Fig. 3 Pareto chart of the standardized effects at $\alpha = 0.05$ concerning (a) arithmetic average roughness; (b) root mean square; (c) maximum profile height.

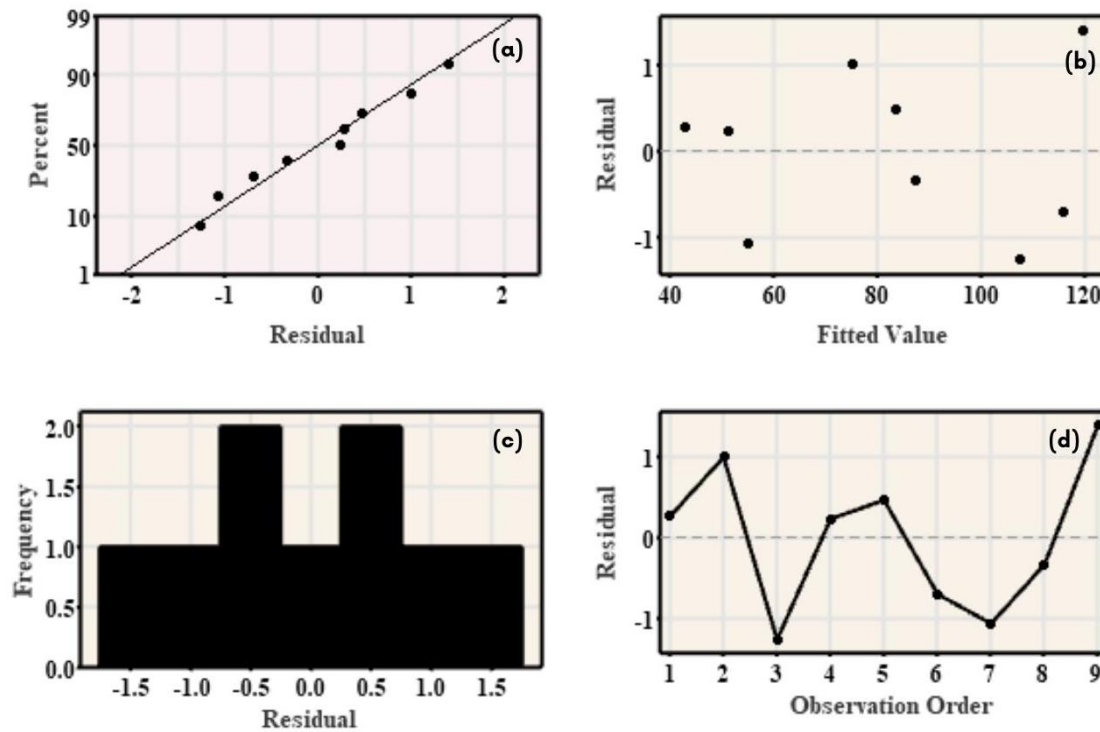


Fig. 4 Residual plots for arithmetic average roughness (Ra) representing: (a) normal probability plots; (b) residual versus fits; (c) frequency histogram; (d) residual versus order.

variation in residuals and the observed order of experimentation and residuals indicated by the histogram, residual versus fit, and residual versus order indicate that there exists a tendency to have run in both positive and negative directions, indicating a strong correlation between observed and predicted values.

3.6 Multivariate analysis

Multivariate analysis is conceptualized by tradition as the statistical study of experiments in which multiple measurements are made on each experimental unit and for which the relationship among multivariate measurements and their structure is important to the experiment's understanding.^[44] In the present study, the surface roughness

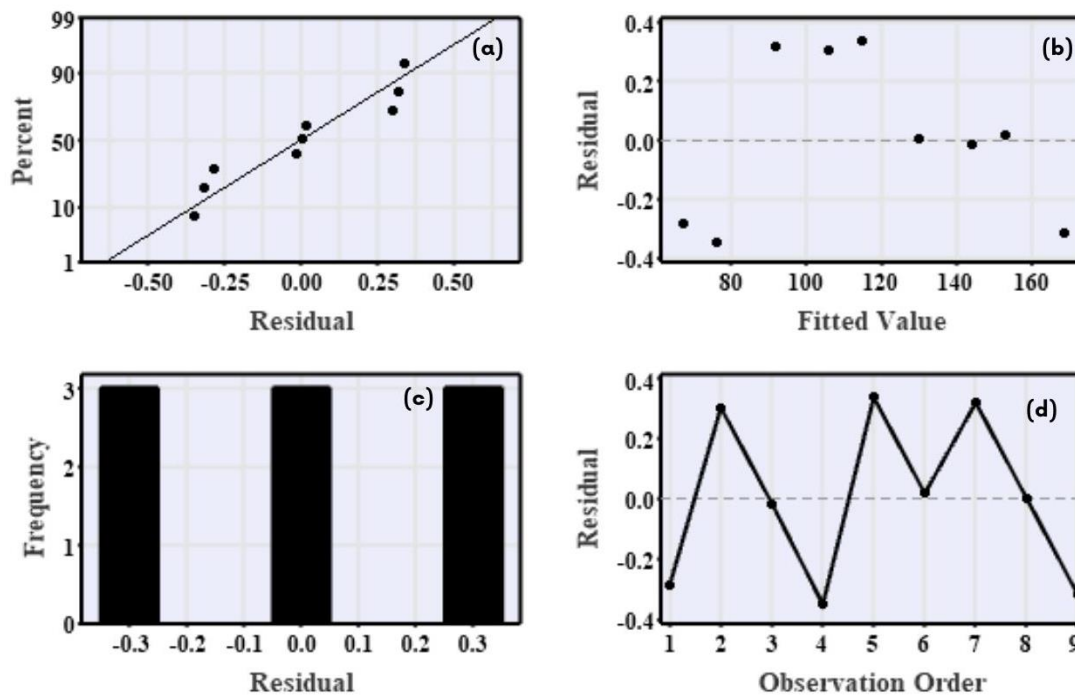


Fig. 5 Residual plots for root mean square (Rq) representing: (a) normal probability plots; (b) residual versus fits; (c) frequency histogram; (d) residual versus order.

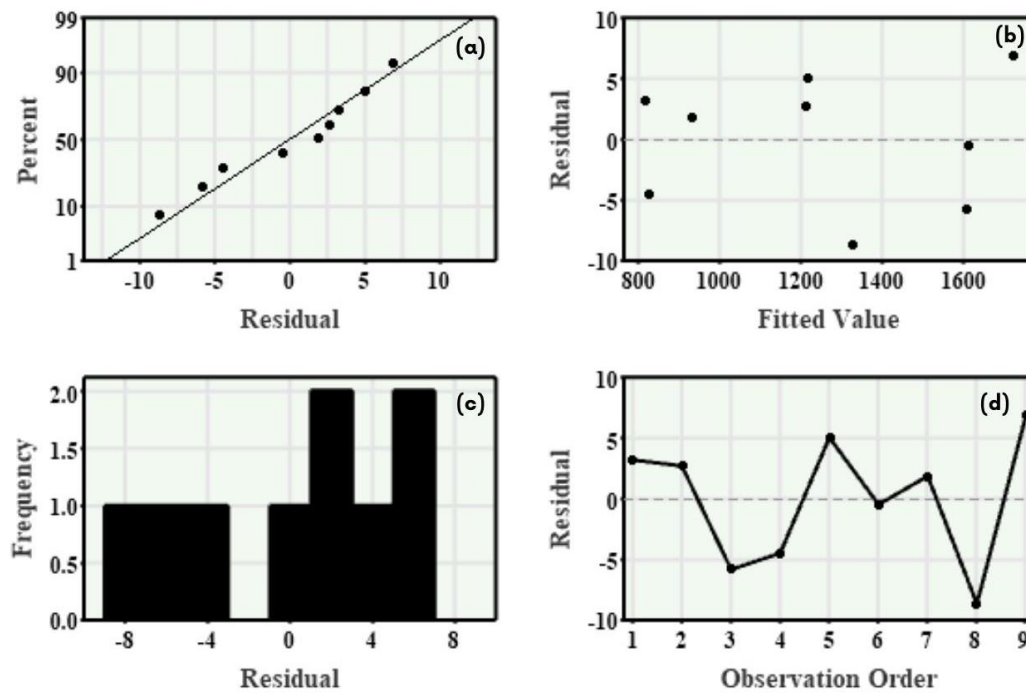


Fig. 6 Residual plots for maximum profile height (Rt) representing: (a) normal probability plots; (b) residual versus fits; (c) frequency histogram; (d) residual versus order.

factors namely arithmetic average roughness, root mean square, and maximum profile height analyzed for the relationship between the conditions and filler content weight percentage. Fig. 7 illustrates the consolidated multivariate analysis chart obtained using MINITAB® 21, wherein the X-axis represents the conditions, Y-axis represents the surface roughness values and the colored legends (circle, square, and triangle) represent the different filler content wt.% (1, 2, and 3%). The results indicate that irrespective of the filler content in the nanocomposites, condition 1 with a sonication time – of 10 min; Compression time – of 5 min yielded the lowest

surface roughness and the trend of increment is seen from conditions 1 to 3. Similarly, it is also observed that irrespective of the pooled effect of the conditions, nanocomposites having 3% filler content showcased the highest surface roughness values.

3.7 Validating experiments

A small set of validating experiments were conducted to check the prediction accuracy of the developed model. The ZnO nanofiller composition was increased to 4, and 5 wt.% in the R_{qexp} , and R_{texp} . Table 11, Table 12, and Table 13 provide the

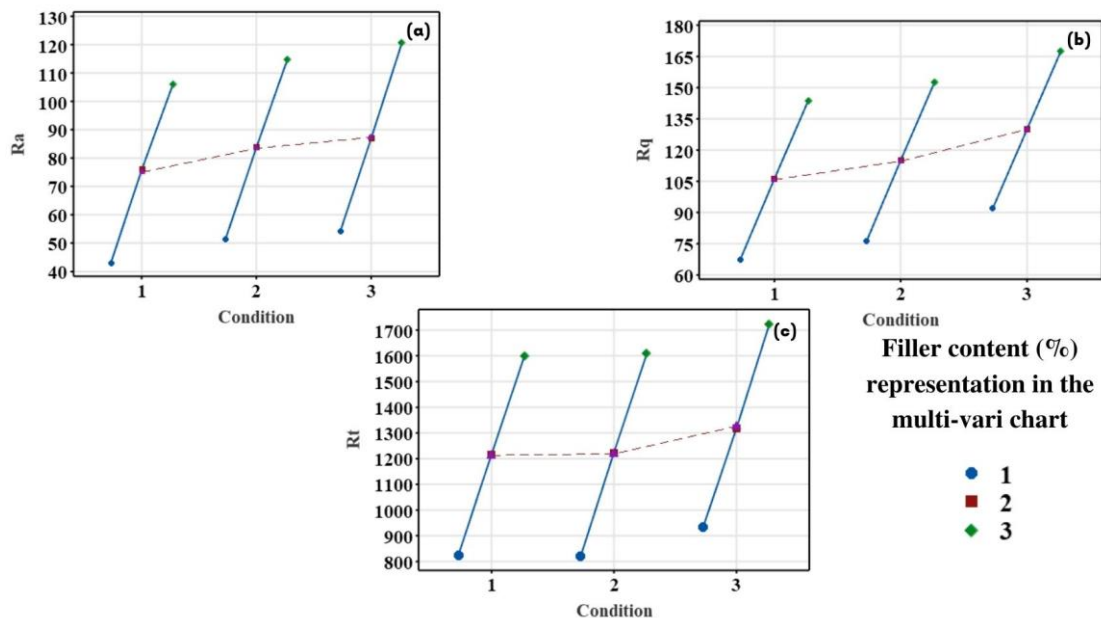


Fig. 7 Multivariate plots for (a) arithmetic average roughness; (b) root mean square; (c) maximum profile height.

Table 10. Amplitude functional parameters of surface roughness measured for EGZ nanocomposites in the validating experiments.

ZnO Weight% in the polymer-filler matrix	Sonication time St (min)	Compression time Ct (min)	Raexp (μm)	Rqexp (μm)	Rtexp (μm)
4	10	5	138	180	1969
4	15	10	146	189	1976
4	20	15	150	203	2073
5	10	5	170	217	2361
5	15	10	178	226	2365
5	20	15	182	242	2462

Table 11. Comparison of experimental and theoretical values for arithmetic average roughness.

ZnO Weight% in the polymer-filler matrix	Sonication time St (min)	Compression time Ct (min)	Raexp (μm)	Rath (μm)	Error%
4	10	5	138	139.538	1.11%
4	15	10	146	147.968	1.35%
4	20	15	150	151.868	1.25%
5	10	5	170	171.805	1.06%
5	15	10	178	180.235	1.26%
5	20	15	182	184.135	1.17%

Table 12. Comparison of experimental and theoretical values for root mean square.

ZnO Weight% in the polymer-filler matrix	Sonication time St (min)	Compression time Ct (min)	Rqexp (μm)	Rqth (μm)	Error%
4	10	5	180	182.335	1.30%
4	15	10	189	191.301	1.22%
4	20	15	203	206.635	1.79%
5	10	5	217	220.652	1.68%
5	15	10	226	229.618	1.60%
5	20	15	242	244.952	1.22%

Table 13. Comparison of experimental and theoretical values for maximum profile height.

ZnO Weight% in the polymer-filler matrix	Sonication time St (min)	Compression time Ct (min)	Rtexp (μm)	Rtth (μm)	Error%
4	10	5	1969	1999	1.52%
4	15	10	1976	2005	1.47%
4	20	15	2073	2112.67	1.91%
5	10	5	2361	2392.5	1.33%
5	15	10	2365	2398.5	1.42%
5	20	15	2462	2506.17	1.79%

comparison of experimental values of amplitude functional parameters of surface roughness with the theoretically calculated value using developed models: R_{ath} , R_{qth} , and R_{tth} through the error percentage. The validating test experiments show that the error is too low between the experimental and theoretically calculated values. Thus, the models could be very well used to predict the surface roughness for any filler weight percentage within the experimental limitation of sonication and compression time values. The current study only looks at the amplitude functional parameters under three different conditions.

3.8 ZnO nanofiller weight percentages

In the future, researchers can focus their efforts on analyzing and constructing prediction regression models for a range of surface topologies and formal solutions by augmenting or varying the different nanofiller materials in the polymer-filler matrix. Moreover, models could also be focused on in the future, considering more parameters that might affect the surface roughness. Developing such models is critical because there is growing interest among researchers and engineers in foreseeing the surface topological performance of nanocomposites during the early design stages without relying

on highly strong material science expertise. By anticipating surface roughness during the early conceptual stages, engineers and researchers can optimize the nanomaterial addition by weight percent for a particular sonication and compression period and ultimately attain the desired quality of the final nanocomposite. On the other hand, the primary advantage of such polynomial regression models is that they may be utilized as inputs to scientific software used by engineers or researchers to perform long-term calculations of surface roughness's amplitude functional parameters. For these reasons, there is always a need to develop such regression models that can reduce the most sophisticated and detailed numerical models to simple polynomial functions that can provide the same information in terms of calculation fidelity while using the least amount of computational memory and effort.

4. Conclusion

The influence of ZnO nanofiller, sonication time, and compression time on the amplitude functional parameters of surface roughness of glass fiber reinforced epoxy-ZnO nanocomposites is examined in this work utilizing atomic force microscopy (AFM) and statistical analysis. The result indicates that increased ZnO nanofiller content in the polymer-filler matrix increases the amplitude functional parameters of the EGZ nanocomposites. Moreover, higher sonication and compression times improved the fineness of the nanocomposite, but the effect of increased nanofiller content in the polymer-filler matrix negated and outweighed them. The analysis of variance (ANOVA) analysis demonstrates that sonication time, compression time, and filler content all have a significant influence on all amplitude functional parameters of surface quality. Nonetheless, the contribution of filler content is substantially more than the combined contribution of sonication and compression time. As a result, the previous conclusion is statistically supported. The developed and validated linear regression equation demonstrates that increasing the ZnO nanofiller content in the polymer-filler matrix by 1 wt.% increases the arithmetic average roughness value by 32.267 μm , the root mean square value by 38.317 μm , and the maximum profile height by 393.50 μm , regardless of the conditional effect.

Conflict of Interest

The authors declare no conflict of interest.

Supporting information

Nor applicable.

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