



## THE EXPERIMENTAL STUDY AND STATISTICAL EVALUATION OF THE WEAR AND HARDNESS OF EPOXY REINFORCED WITH NATURAL MATERIALS

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**Abstract:** This work focuses on study the effect of Date Seeds (DS) and Olive Seeds (OS) on wear rate and hardness property of epoxy resin. Olive and dates seeds were added to epoxy at weight fraction (0, 8, 13, & 18wt%) with grain size (300, 450 & 600 $\mu$ m). The composite specimens were prepared by Hand-Lay up technique and cut according to standard test. The results show that the hardness of composites increases with weight fraction of powder increase and the wear rate decreases with increasing weight fraction of powder. Also it is found that the higher value of hardness and wear resistance were at (% =18 wt%) with grain size (300 $\mu$ m) for specimens reinforced with olive seeds. The mathematical model results show that the weight fraction of powder have higher effect than particle size on properties.

**Keywords:** Composite materials, natural materials, wear test, hardness test, mathematical model.

### الدراسة العملية والتقييم الاحصائي للبلى والصلادة لمادة الايبوكسي المدعم بمواد طبيعية

**الخلاصة:** يتركز هذا العمل على دراسة تأثير مسحوق نوى الزيتون والتمر على خاصية الصلادة ومعدل البلى لمادة الايبوكسي. تم اضافة مسحوق نوى الزيتون والتمر الى الايبوكسي بالنسب الوزنية التالية (0, 8, 13, 18 wt%) مع حجم حبيبي (300, 450&600 $\mu$ m). حضرت العينات المتراكبة بطريقة القولبة اليدوية ثم تم اجراء تقطيع العينات حسب المواصفة القياسية لكل فحص. بينت النتائج بان الصلادة للمتراكبات تزداد بزيادة الكسر الوزني للمسحوق ومعدل البلى يتناقص مع زيادة الكسر الوزني للمسحوق. أيضاً وجد بان اعلى قيم صلادة ومقاومة بلى كانت عند نسبة وزنية (18 wt%) وبجسم حبيبي (300 $\mu$ m) للمتراكبات المقواة بمسحوق نوى الزيتون. بينت نتائج الموديل رياضي بان الكسر الوزني لمادة التقوية كانت ذات تأثير أكثر من الحجم الحبيبي على الخواص.

### 1. Introduction

Wear was defined as damage to a solid surface, generally involving progressive loss of material, due to relative motion between contacting surfaces. The five main types of wear were abrasive, adhesive, fretting, erosion and fatigue wear, which were commonly observed in practical situations. Abrasive wear was the most important among all the

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forms of wear because it contributes almost (63%) of the total cost of wear. Abrasive wear was caused due to hard particles or hard protuberances that were forced against and move along a solid surface [1].

The wear properties can be varied substantially through changes in the microstructure, the morphology, volume (or weight) fraction and mechanical properties of the reinforcing phase, and the nature of the interface between matrix and the reinforcement. Modern composite materials are usually optimized to achieve a particular balance of properties for a given range of applications. Given the vast range of materials that may be considered as composites and the broad range of uses for which composite materials may be designed, it is difficult to agree upon a single, simple, and useful definition [2].

In the last decade, a growing interest in the use of vegetable reinforcements in the development of composite materials has gained momentum. Lignocellulosic reinforcements present interesting features such as low cost, low density, and besides they are biodegradable and non-abrasive [3].

Olive seeds, a lignocellulosic material, is currently used mainly as energy source. Recently, the literature references presented use as a raw material to produce activated carbon and furfural and as heavy metal biosorbent [4].

These materials thus form inexpensive “new or secondary resources”, which could make them more valuable for wider utilization. When such materials are used in composites, developing countries, which produce these, become part of global composite industry as developer and manufacturer leading to increasing revenues and the creation of jobs [5&6].

Dwivedi & Chand in (2008) have studied influence of wood flour loading on tribological behavior of epoxy composites, reports that the reinforcement of wood flour increases the load carrying capacity of epoxy and decrease its wear resistance [7].

Qutaiba in (2011) has studied of a new range of sustainable reinforced polymer composite materials using powdered olive pits as a novel filler material to be used with synthetic resin. Also the influence of the untreated and treated powder loading (weight fraction) on the void content and the mechanical properties of the composites was examined. It was found that significant improvements in mechanical properties of composites reinforced with treatment of olive pits powder than composites reinforced with un treatment olive pits [8].

Noori & et al in (2013) have studied the wear and hardness for epoxy reinforced with nano and micro  $\text{TiO}_2$ . It was found that the wear rate increased with the load applied and decreased with increasing weight percentage of titanium dioxide [9].

Hamma, A. A. & et al in (2013) have studied the effects of date stone flour (DSF) on morphology, thermal, and mechanical properties of polypropylene (PP) composites in the absence and presence of ethylene-butyl acrylate-glycidyl methacrylate (EBAGMA) used as the compatibilizer. The study showed through scanning electron microscopy analysis that EBAGMA compatibilizer improved the dispersion and the wettability of DSF in the PP matrix. Thermogravimetric analysis (TGA) indicated a slight decrease in the decomposition temperature at onset (Tonset) for all composite materials compared to PP matrix, whereas the thermal degradation rate was slower. Differential scanning

calorimetry (DSC) data revealed that the melting temperature of PP in the composite materials remained almost unchanged. The nucleating effect of DSF was however reduced by the compatibilizer. Furthermore, the incorporation of DSF resulted in the increase of stiffness of the PP composites accompanied by a significant decrease in both the stress and strain at break. The addition of EBAGMA to PP/DSF composites improved significantly the ductility due to the elastomeric effect of EBAGMA [10]. The aim of this research was to study the influence of weight fraction and particle size of powder on wear and hardness properties of Epoxy reinforced by olive and dates seeds.

## 2. Theoretical Analysis

Wear is one of the most commonly encountered industrial problems leading to the replacement of components and assemblies in engineering. In general there is great enthusiasm for wear resistant of the polymer, in order to obtain the optimal wear rate without compromising the beneficial properties of the matrix material [11].

The following relation is used to investigate the wear rate which is [12]:

$$W.R = \frac{\Delta W}{S.D} \quad (1)$$

Where:-

W.R:- Sliding wear rate is the weighted (gm/mm).

$\Delta W$ :- The change in weight during the experiment and calculated from following relationship:-

$$\Delta W = W_1 - W_2$$

Where:-

$W_1$ :- Weight of sample before the test (gm).

$W_2$ :- Weight of sample after the test (gm).

S.D: a sliding distance calculated from the following low:

$$S.D = S * t \quad (2)$$

Where:

S:- the speed of sliding (mm/min).

t:- time slip (min).

## 3. Mathematical Model

Response surface method (RSM) is a collection of a mathematical and statistical technique that are useful for modeling, analysis and optimizing the process in which

response of interest is influenced by several variables and the objective. In many engineering fields, there is a relationship between an output variable of interest "z" and a set of controllable variables  $\{x, y, \dots, N_n\}$ . In some systems, the nature of relationship between y and x values might be known. Then, a model can be written in the form [13]:

$$z = f(x, y, \dots, N) + \mathcal{E} \quad (3)$$

The variables (x, y and N) are independent variables where the response (z) depends on them. The experimental error term, denoted as  $\mathcal{E}$ . In this study, multiple polynomial (least square fitting) regression analysis is used to establish a mathematical model among the experimentally obtained parameters. Multiple regression analysis techniques are applied to relate the weight and grain size of powder for two types of powder (dates seeds and olive seeds), the best form of the relationship between the property, weight and grain size of powder parameters is chosen in the form of [14]:-

$$z = a_0 + b * x + c * y + d * x^2 + e * y^2 + g * x * y \quad (4)$$

The analysis of variance (ANOVA) is also called coefficient of multiple determination referred to ( $R^2$ ) measure the proportionate reduction of total variation in associated with use of the set of predictors in the model (is used to check the validity of the model) it is defined in terms of SST, SSR, and SSE as [15]:

$$R^2 = \frac{SSR}{SST} = 1 - \frac{SSE}{SST} \quad (5)$$

Where:

SSE:- the sum of squared error.

SSR:- the regression sum of squares.

SST:-the total correct sum of squares.

For the three factors, the second polynomial (regression) could be expressed as:

$$Z = b_0 + b_1 * X + b_2 * Y + b_3 * N + b_4 * X^2 + b_5 * Y^2 + b_6 * N^2 + b_7 * X * Y + b_8 * X * N + b_9 * Y * N \quad (6)$$

Where:

$b_0$ :- is the free term.

$b_1, b_2, b_3$ :- are coefficients linear terms.

$b_4, b_5, b_6$ :-are quadratic terms.

$b_7, b_8, b_9$ :- are interaction terms.

#### 4. Experimental Work

The polymeric composite specimens made from epoxy as a matrix and natural powder: olive and dates seeds that grinded by using (JF-SD-100 Pulverize) as a reinforcement with different weight fraction of powder (0, 8, 13 & 18 wt%) and sieved to

the required particle size (300, 450&600 $\mu\text{m}$ ).Then the wear and hardness were performed to determine the characteristics of the studied composite. The type of epoxy resin is (Swiss cherry) provided from the Saudi Arabia Company in the form of transparent viscous liquid at room temperature. Epoxy and hardener used in this study in ratio of 3:1.

## 5. Preparation of Composites

The composite specimens were fabricated by using hand lay-up technique. Composites having different powders content were prepared by varying the type and weight for olive seeds and dates seeds powders. In the first process of preparing the composite specimens' preparation process is to set the weight and particle size of powders content in the composite.

The amount of resin needed for each category of composite is calculated after that. Then the resin was mixed uniformly with hardener, the mixture was poured carefully into the moulds made of stainless steel and left in the mould for 24 hours. After the composites were fully cured, they were demoulded, and then put the specimens in oven at (55 °C) for (1 hrs) [16].

Specimens are prepared after the composites are ready. For hardness test the specimens are cut according to (ASTM DI-2242) standard. Samples have been cut into a diameter of (40mm) and a thickness of (5mm) [17].

The hardness test is performed by using hardness (Shore D). For each specimen five hardness measurements were taken and the average hardness is calculated.

The rotating Pin -on- Disc wear testing machine done within a conditioned at room temperature. The weighing method was used to determine the mass loss of the test specimens before and after the test. The sample was fixed in holder and abraded under load (0.5 Kg) and a sliding speed (2 m/sec.) with different time (1, 2& 3 min). The dimension of the specimen was 9.5 mm diameter and 20 mm length based on the standard wear tests described in ASTM standard D5963-97a [18].

## 6. Results &Discussions

### 6.1. Hardness Test

Fig. (1&2) show the relationship between hardness and weight fraction of olive and dates seeds composites. The results had revealed that the hardness increases with increasing weight fraction of particles because of increasing wettability or bonding (interaction) between the matrix and the powder particles, But the value of hardness decrease when the particle size increasing due to the composite become brittle due to the large content.

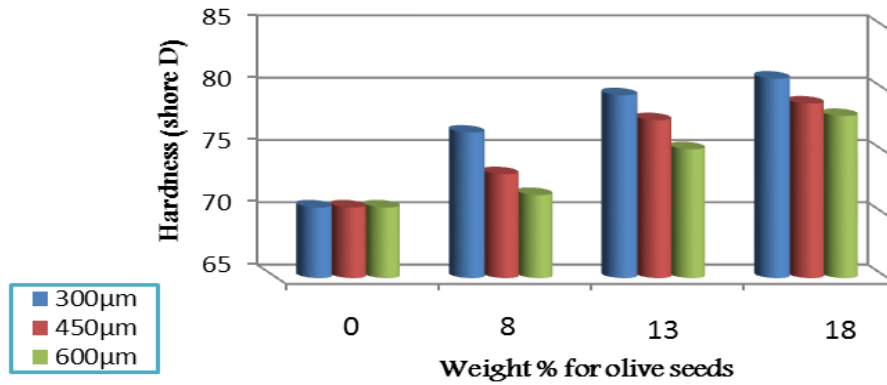


Figure (1): The relationship between hardness values & wt% for composite reinforced with olive seeds powder as a function of particle size.

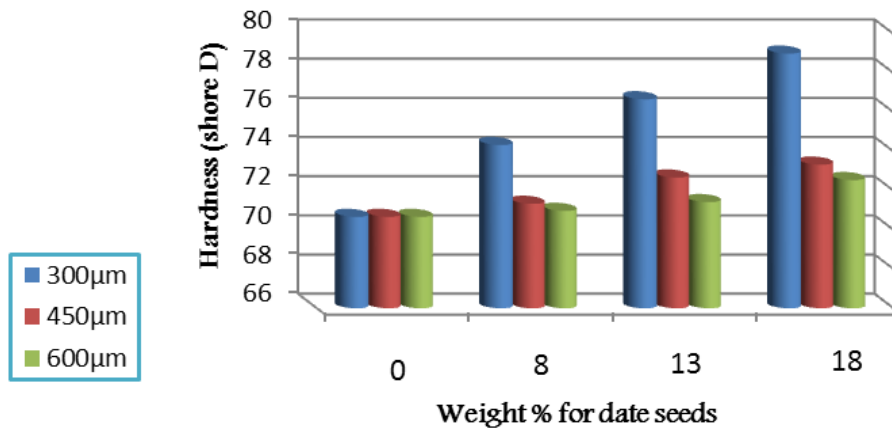


Figure (2): The relationship between hardness values & wt% for composite reinforced with dates seeds powder as a function of particle size.

## 6.2. Wear Rate

The effect of time for applied normal load on wear rate for composite specimens reinforced with olive and dates seeds are illustrated in Fig. (3-8). It can be clearly from these figures that the increasing times of applied load the wear rate of composite specimens increasing. The wear rate of the pure epoxy is more than that of the reinforced epoxy for all time of load applied. This is due to presence of reinforcing powder particles that lead to increase the average hardness of the composite. Moreover, as the percentage of reinforcement increases, the wear rate of the composite decreases. Also from these figures it can be seen that the wear rate is increasing with increase particle size of powders. This is may attributed at coarser particles, the depth of penetration of the abrasive particle is so high that a large portion of materials is removed from the specimen surface leaving behind large cavities in the worn surface. It is found that the results obtained from composite reinforced with olive and dates seeds powder has a lower value of wear rate obtained at composite reinforced with olive seeds powder at (=18 wt%) with particle size (=300µm).

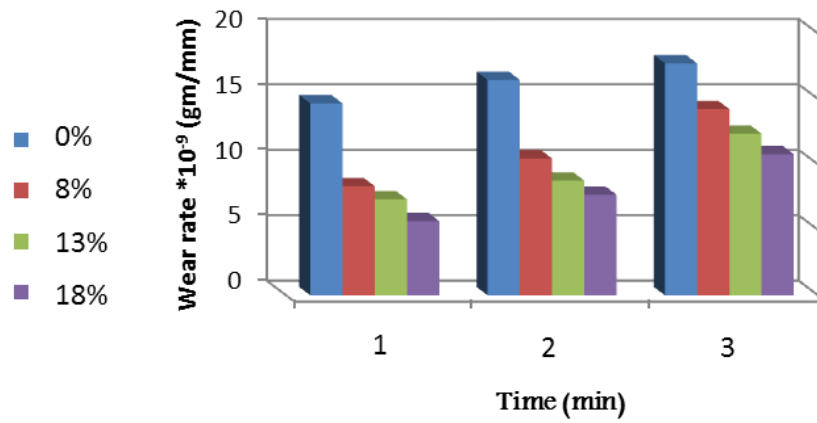


Figure (3): Plots between wear rate and time for composite reinforced with olive seeds powder at (300µm) as a function of powder content in composite.

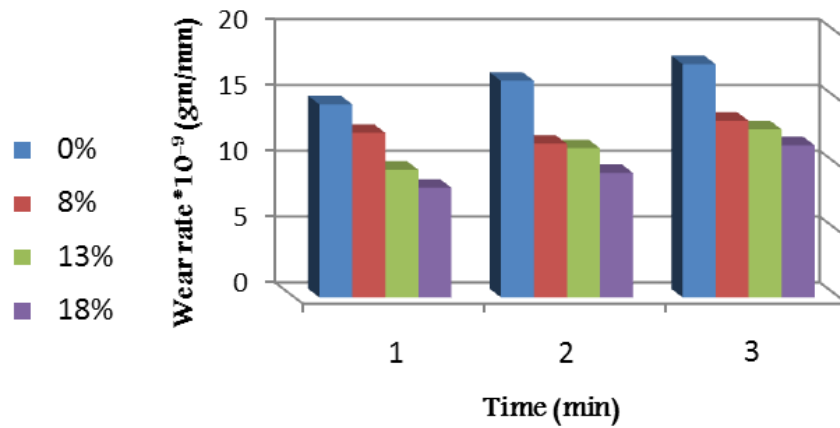


Figure (4): Plots between wear rate and time for composite reinforced with olive seeds powder at (450µm) as a function of powder content in composite.

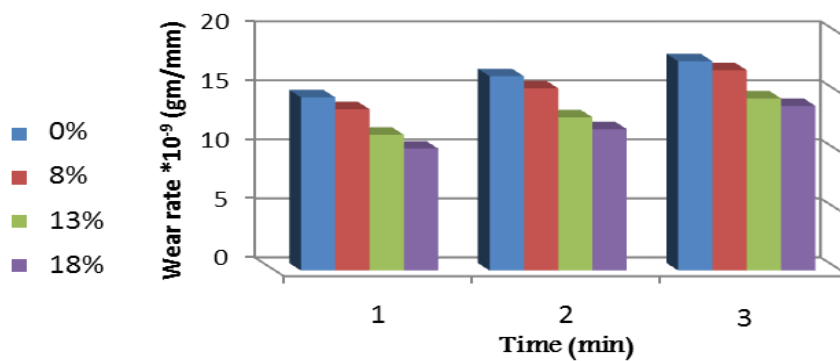


Figure (5): Plots between wear rate and time for composite reinforced with olive seeds powder at (600µm) as a function of powder content in composite.

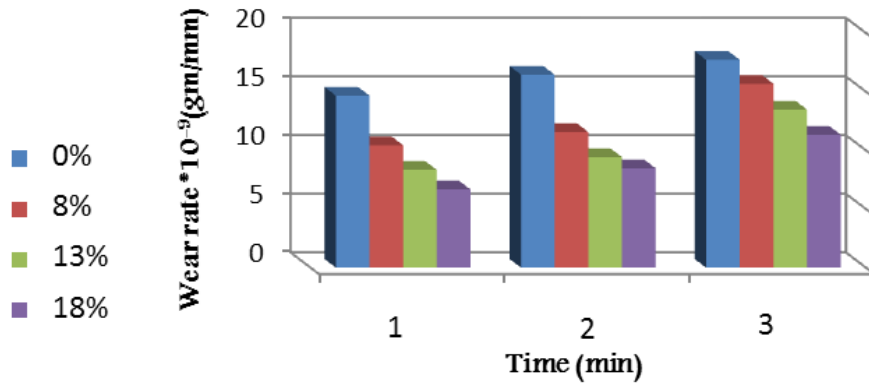


Figure (6): Plots between wear rate and time for composite reinforced with dates seeds powder at (300µm) as a function of powder content in composite.

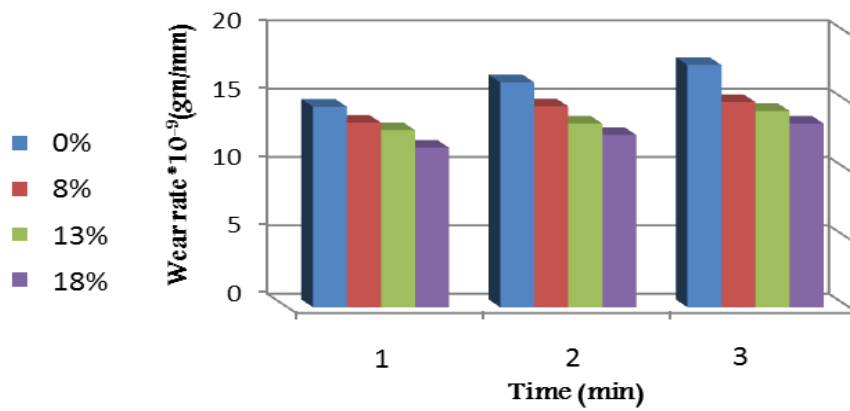


Figure (7): Plots between wear rate and time for composite reinforced with dates seeds powder at (450µm) as a function of powder content in composite.

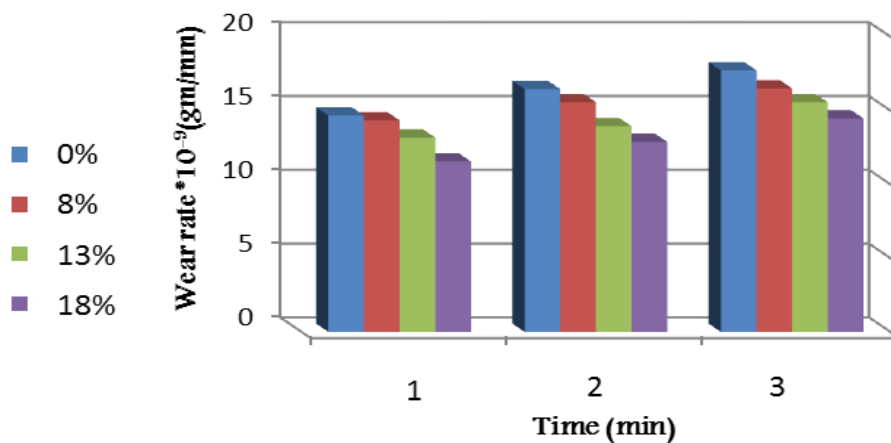


Figure (8): Plots between wear rate and time for composite reinforced with dates seeds powder at (600µm) as a function of powder content in composite.

### 6.3. Mathematical Model

The experimental results are modeled using RSM. Table (1) shows the summary of models and coefficient multiple determinations ( $R^2$ ) of the properties as function of ( $x=$



weight fraction of powder) and (y=particle size) where (z = represent the hardness value). It can be seen from these models that the weight percentage of powder have greater effect than the particle size on the properties.

Table (1): Mathematical model results for hardness of epoxy reinforced by olive and dates seeds.

property	Mathematical model and coefficient of multiple determination	Figure
Hardness value for composite reinforced with olive seeds	$Z=72.757+(0.7722)*x+(-0.002113)*y+(-0.00694)*x^2+(-0.00001)*y^2+(-0.00027)*x*y$ $R^2=0.9825$	
Hardness value for composite reinforced with dates seeds	$Z=79.487+(0.9439)*x+(-0.04561)*y+(0.00284)*x^2+(0.0000562)*y^2+(-0.00181)*x*y$ $R^2=0.995$	

#### 6.4. Development of Mathematical Model Results of Wear Test

The validity of regression models developed is further tested by drawing scatter diagrams. Typical scatter diagrams for all the models are presented in Table (2). Where (X= weight fraction of powder, Y= particle size & N= time). The observed values and predicted values of the responses are scattered close to the (45°) line, indicating an almost perfect fit of the developed empirical models [19].

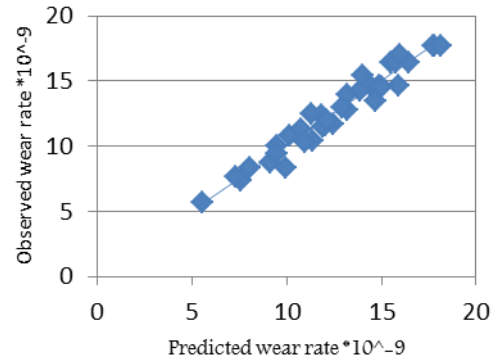
Table (2): Mathematical model results for wear of epoxy reinforced by olive and dates seeds.

property	Mathematical model and coefficient of multiple determination	Figure
Wear rate for composite reinforced with dates seeds	$Z=3.646-0.627*x+0.041*y+1.982*n+0.00022*x^2-0.00004*y^2+0.236*n^2+0.00078*x*y+0.00112*x*n-0.00316*y*n$ $R^2=0.9712$	

Wear rate for composite reinforced with olive seeds

$$Z = 12.5333 - 0.838 * x - 0.00073 * y + 1.358 * n + 0.0064 * x^2 + 0.00001 * y^2 + 0.319 * n^2 + 0.00071 * x * y + 0.0273 * x * n - 0.0026 * y * n$$

$$R^2 = 0.977$$



## 7. Conclusions

The main conclusions of results were:-

1. The composite specimens reinforced with olive seeds gives higher hardness and lower wear rate values than composites specimens reinforced with dates seeds.
2. Higher value of hardness and wear resistance obtained at wt% =18% and particle size=300 $\mu$ m for two types of powder.
3. Hardness mathematical model results show that the weight fraction of powder have higher effect than powder size on properties.
4. Development mathematical model for wear test shows that the observed values and predicted values of the responses are scattered close to the (45°) line.

## 8. References

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