



# Journal of Materials and Engineering Structures

## Research Paper

### Trapping of oil in an epoxy-based polymer matrix with activated carbon and its effect on tribological behavior

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#### ARTICLE INFO

##### Article history :

Received : 19 September 2019

Revised : 11 December 2019

Accepted : 18 December 2019

##### Keywords:

Self-lubricating

Polymers

Wear

Friction

#### ABSTRACT

The incorporation of lubricating oil at high levels in the epoxy composite materials makes it possible to obtain materials with low friction and wear. This article evaluates the wear behaviour of an epoxy-based composite obtained by the addition of lubricating oil and activated carbon. An oil content of up to 31wt% is trapped and maintained in the matrix by the presence of activated carbon. The wear experiments are carried out on a pin-on-disk type tribometer. The tests are carried out on pins of different compositions sliding on steel discs (XC48) under dry friction conditions. The effect of various parameters such as speed and loading has been investigated. The wear surfaces are analyzed by using a scanning electron microscope to observe the different wear mechanisms. The results show that the addition of activated carbon made it possible to trap large quantities of oil in the matrix. This decreased greatly the coefficient of friction and increased the capacity of the sample to withstand higher values of sliding velocities and pressures.

## 1 Introduction

Composite materials offer many advantages such as ease of manufacture, lightness, low cost and excellent performance. As tribological materials, composites are very much interesting for machinery components and various mechanical devices as gaskets, gears, bearings, brakes and clutches, drive belts, cams, wheels, piston rings ... etc [1, 2].

As a result, the wear behaviour and the contact resistance of polymers and composite polymers used in mechanical parts have been the subject of much research [3–12]. These studies have demonstrated that the wear behaviour of these materials is different from that of traditional metallic materials, and that incorporation at low mass percentage of certain additives such as carbon fibers, carbon nanotubes, and powder graphite, PTFE, etc. can have a beneficial effect on the

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tribological properties of polymers. The influence of these solid lubricants depends on their intrinsic properties, size and hardness of the particles [13, 14].

The incorporation of liquid lubricants such as oil to polymers is another approach that improves the tribological performance of epoxy composites [15, 16]; the lubricating effect of the oil released, trapped in the resin, offers an epoxy resin with a low coefficient of friction and a low wear rate. These solid and liquid lubricants can be incorporated into the polymers as particulate fillers and provide them intrinsic lubricating properties.

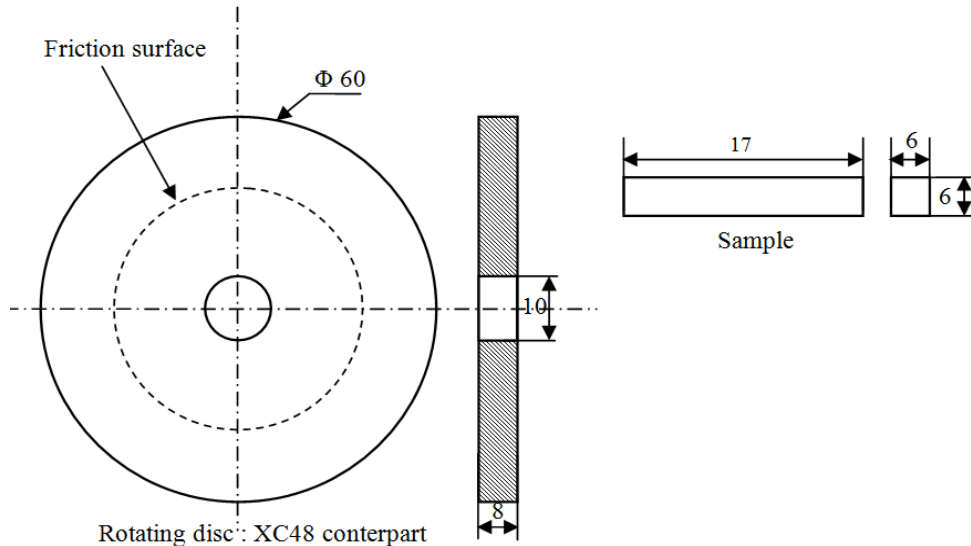
In the present work, a lubrication approach by incorporating high percentage lubricating oil into the polymer matrix has been considered. Our results show that the addition of oil in the polymer matrix is limited to a level of 22w% beyond which the oil is rejected by the matrix.

A solution for increasing this oil content up to 31w% is the addition of active carbon into the polymer matrix. Activated carbon is characterized by the presence of a large number of open micro pores which act as adsorption sites [17]. Probably as a result of this characteristic, the activated carbon makes it possible to trap and maintain this level of oil in the matrix, a level from which the matrix is saturated and the polymerization cannot be achieved.

## 2 Materials and methods

### 2.1 Materials and sample preparation

The wear tests are performed on a pin-on-disc type tribometer designed and manufactured at the University of Tizi-Ouzou. The prismatic shaped pins (36 mm<sup>2</sup> section and 17 mm length) are obtained from epoxy plates loaded at different percentages of additives in oil mass and powdered activated carbon. These plates are obtained by molding then cut and milled in parallelepipedic form with the same cutting conditions. The disks (60 mm diameter and 8mm thickness), are used as conterface material, these disks are obtained by turning from hardened XC48 steel rounds, roughness (Ra = 0.12).



*Fig. 1 – Forms and dimensions of Pin and disk*

The preparation of the composite plates, with different compositions, is obtained with an epoxy resin matrix (type R123, bisphenol A / F epoxy resin) and a hardener (type R614) supplied by soloplast-vosschemie. The low density activated carbon (CAS: 7440-44-0) added to the resin matrix is provided by biochem chemopharma. The lubricating oil used is Singer brand.

The mixtures are then homogenized one after the other using an agitator equipped with two stainless steel propellers. After homogenization, the mixtures are poured into the corresponding molds previously prepared.

After polymerization and demoulding, the plates are cut to the required dimensions using a chainsaw. Four different types of compositions have been prepared:

- Epoxy (named as Ep)
- Epoxy/Activated carbon (8 wt %) (named as Ep/Ca)
- Epoxy/Oil (22 wt %) (named as Ep/Oil)
- Epoxy/ Activated carbon (6 wt %) /Oil (31 wt %) (named as Ep/Ca/Oil)

Addition of the lubricating oil into the matrix at different percentages by weight is made by adding oil little by little to figure out the maximum oil percentage that the matrix can hold. We concluded that 22w% of the exceeding oil is rejected by the matrix; the excess oil rises to the surface of the composite after polymerization (Fig. 2). This means that the matrix retains only oil levels below 22w%.



**Fig. 2 – Excess oil rises to the surface after polymerization of the composition Ep/Oil**

To increase this oil percentage in order to further reduce the coefficient of friction, an oil entrapment which reaches 31w% in the polymer matrix is obtained by the addition of 6w% activated carbon. A value of 6 w% of activated carbon is sufficient to trap approximately 31 w% oil. Below 6 w%, the trapping of oil in the matrix does not reach 31w% oil. Moreover, below 31w% oil the composite does not improve tribological characteristics. Other tests elaborated with 40w% oil and 10w % activated carbon present a bad polymerization. A complete polymerization is ensured with a concentration of 31w% of oil.

## 2.2 Tribological tests

In order to determine the tribological characteristics of the epoxy-based composites, tests under dry friction conditions are carried out using a pin-on-disk type tribometer equipped with an electric motor with variable speed drives. We have used a strain gauge sensor to measure simultaneously and independently the normal force and the tangential force during the wear process. The sensor is equipped with two Wheatstone bridges. The design of the sensing element allows to measure separately the normal force and the tangential force. The four strain gauges (G1 G2 G3 G4) measure only the normal force, the effect of the tangential force cancels out automatically thanks to the alternation of the signs in the output voltage of Wheatstone bridge (equation 1). The same can be for the measurement of the tangential force.

$$V_{out} = \frac{V_{in}}{4} \left( \frac{\Delta R_1}{R_1} - \frac{\Delta R_2}{R_2} + \frac{\Delta R_3}{R_3} - \frac{\Delta R_4}{R_4} \right) \quad (1)$$

R: Electrical resistance of the gauge

The measurement of the parameters is made using a data acquisition system; the results are read directly on the computer screen in real time. A representation of the experimental setup of friction tests is given in Fig. 3.

Under dry sliding conditions, the disk rotates and rubs against the composite pin for one hour at room temperature; the sliding speed and the contact pressure are kept constant at 1m/s and 1MPa, respectively

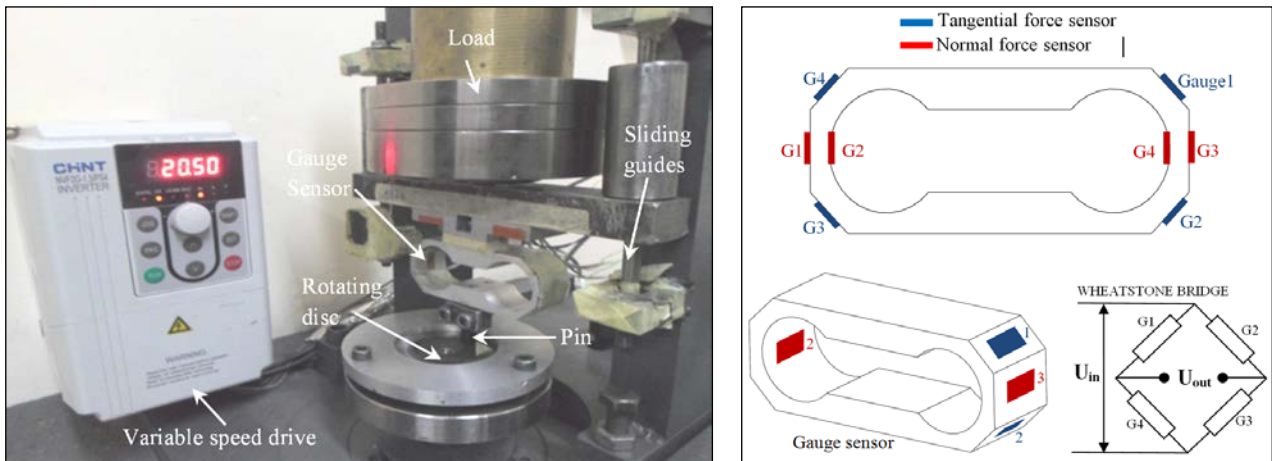


Fig. 3 – Pin-on-disk type tribometer

### 3 Results and discussion

#### 3.1 Coefficient of friction

The tests are carried out at room temperature under controlled load and sliding velocity conditions. For each test, we record the graphs of the normal force  $F_N$  and the tangential friction force  $F_T$  as a function of time. The evolution of the coefficient of friction as a function of time is deduced from the graphs of  $F_N(t)$  and  $F_T(t)$  by carrying out the ratio  $F_T(t) / F_N(t)$ . At least four tests have been realized for each composition. For a better comparison of the friction coefficients of the four compositions, all graphs are given in Fig. 4; we have selected the curves showing the reproducibility of the results for each composition during the wear tests.

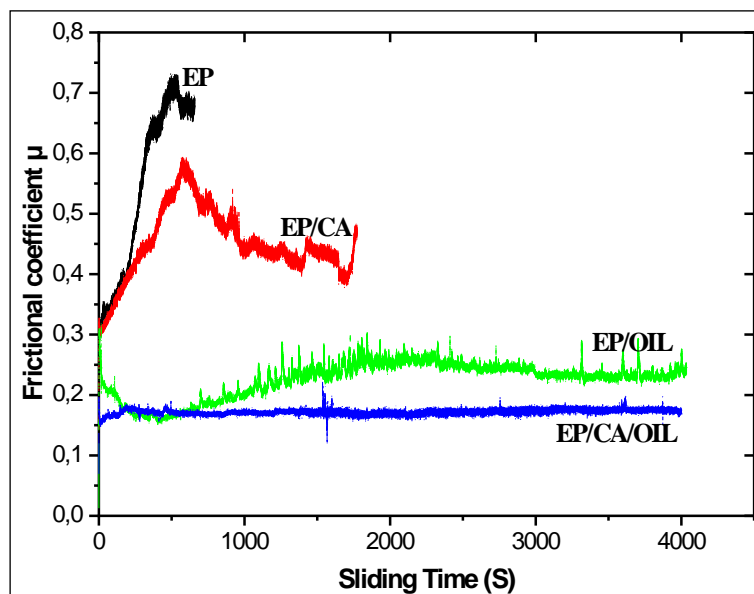
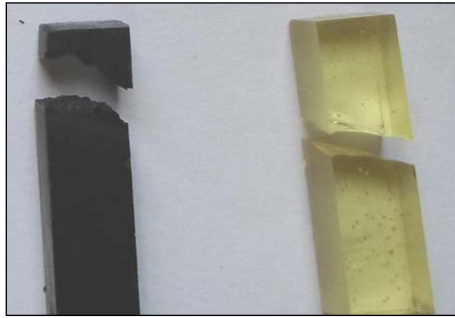


Fig. 4 – Coefficient of friction for the four compositions

The coefficient of friction of the composition Ep reaches values around 0.7. The composition Ep/Ca, which is a charged composition of activated carbon, has a coefficient of friction of 0.45. As shown in Fig. 4, the wear tests are stopped for the compositions (Ep and Ep/Ca) because of the deformation and the breaking of the pins during the tests (Fig. 5).



**Fig. 5 – Samples Ep and Ep/CA broken during a wear test at 1Mpa.m/s**

The Ep/Oil composition has a remarkable improvement in the coefficient of friction which reaches 0.24. But after one hour of running time under standard wear conditions, the composite begins to undergo plastic deformation following the increase in temperature (Fig. 6).



**Fig. 6 – A sample Ep/Oil deformed during a wear test at 1Mpa.m/s**

In order to improve the coefficient of friction and the composite PV product (product of the normal pressure and the sliding velocity), a high concentration of oil in the matrix is necessary. The composition Ep/Ca/Oil with 31w% oil content improves the coefficient of friction from 0.7 for pure epoxy to 0.16 for our composition. The PV factor reaches a value of 3MPa.m/s for which the pin composite begins to undergo plastic deformation (Fig.7).



**Fig. 7 – A sample Ep/Ca/Oil deformed during a wear test at 3Mpa.m/s**

The improvement in the coefficient of friction and PV factor is due to the addition of activated carbon which allows the trapping of oil at large quantities in the matrix.

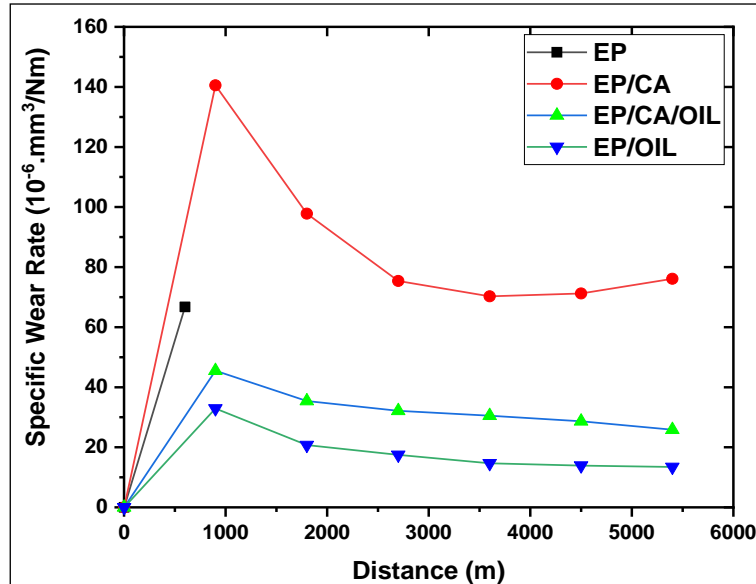
### 3.2 Wear rate tests

To quantify the mass loss and the wear rate, the samples (pins) are weighed before and after each experiment using an electronic balance with an accuracy of  $10^{-5}$  g. The specific wear rate of the material is calculated using equation (2).

$$\omega_s = \frac{\Delta m}{\rho \cdot F_N \cdot L} \left( \frac{\text{mm}^3}{\text{Nm}} \right) \quad (2)$$

In which  $F_N$  is the normal load applied to the sample during sliding,  $\Delta m$  is the mass loss of the sample,  $\rho$  is the density of the sample, and  $L$  is the total sliding distance.

The wear tests for the Ep/Oil and Ep/Ca/Oil compositions are carried out under standard sliding conditions of 1 MPa and 1 m/s. For the Ep and Ep/Ca compositions, the tests are carried out at a sliding speed  $V = 1\text{ m/s}$  and a pressure  $P = 0.5\text{ Mpa}$ , in order to avoid breaking of the samples. Fig. 8 gives the specific wear rate graphs at 5500 meters for the four compositions. One test is run for each composition; Fig. 8 presents the evolution of wear rate during the wear process.



*Fig. 8 – Specific wear rate for the four compositions*

We deduce that for the studied compositions Ep/Ca, Ep/Oil and Ep/Ca/Oil, the rate of wear decreases with the sliding distance. For composition Ep, due to the breakage of the sample after a distance of 800 meters, we are unable to record a sufficient number of points to obtain a wear rate graph.

The Ep and Ep/Ca compositions have a high wear rate. The morphology of the worn surfaces of the pins is given in Fig. (10a, 10b). The images show that the worn surface is smooth; the adhesive wear acts as the main wear mechanism which leads to the tearing of the material and the breaking of the samples. For the Ep/Ca composition, under the wear conditions used, the activated carbon added alone is not a good solid lubricant.

However, after the addition of oil, the compositions Ep/Oil and Ep/Ca/Oil give a wear rate of  $15 \cdot 10^{-6}$  and  $25 \cdot 10^{-6}$   $\text{mm}^3/\text{Nm}$  respectively. These compositions have a better wear resistance compared to the Ep and Ep/Ca compositions. During wear the lubricating oil is released and forms a thin layer on the contact surface (Fig. 9), this oil film helps to separate the two contacting surfaces, thus reducing wear by adhesion.

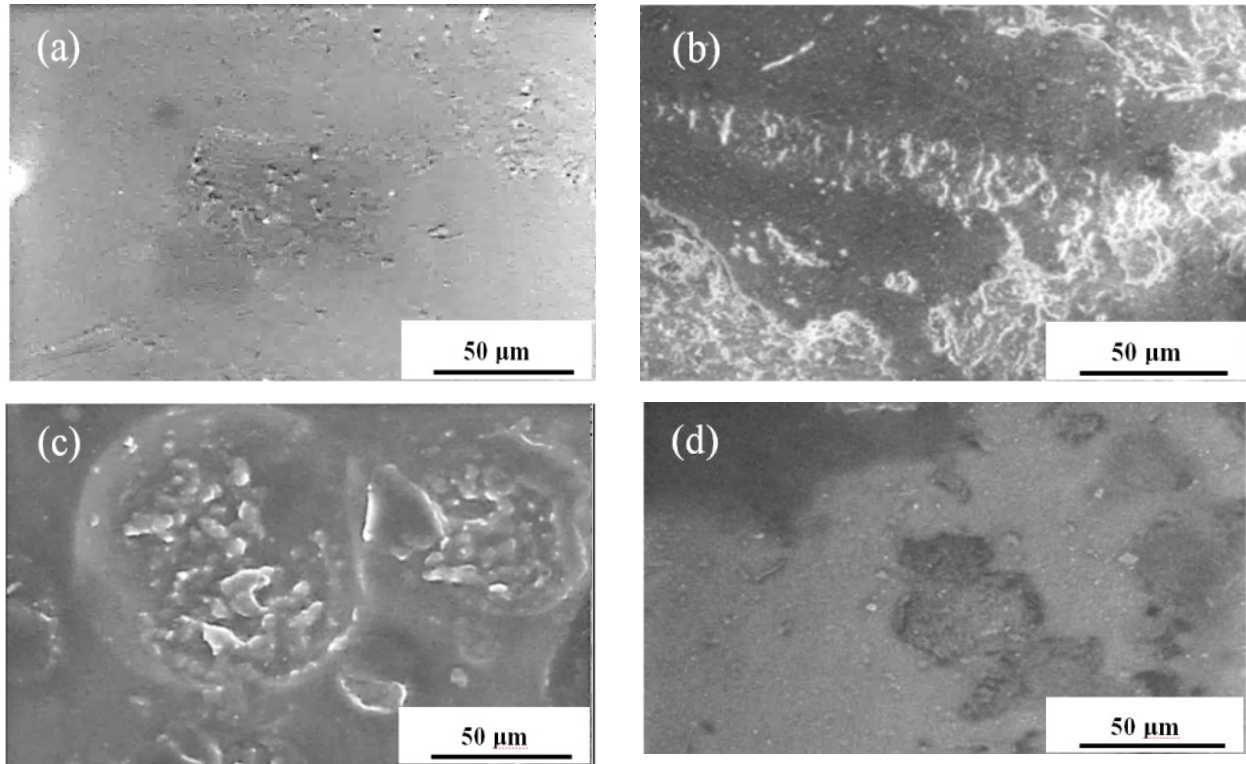


*Fig. 9 – Thin layer on the contact surface of the pin (Ep/Ca/Oil) on disc*



As shown in Fig. (10d), the wear of the Ep/Ca/Oil composition is probably due to a wear mechanism mainly caused by the active carbon wear debris which forms a third body thus causing abrasive wear.

For the Ep/Oil composition, the lubricating effect of the oil released during the friction causes a reduction in the coefficient of friction. Fig. (10c) shows that wear debris is trapped in cavities left by oil bubbles. Thus, reducing the amount of wear debris weakens the abrasive effect of wear debris as a third body in the contact area. This explains the decrease in the specific wear rate shown in Fig. 8.



**Fig. 10 – MEB of pins contact surface for each composition at 1m/s sliding speed and 1MPa contact pressure**

The addition of graphite has a minor influence on the wear rate (Fig. 8). In addition, there is a significant gain which is the reduction of the friction coefficient, and the remarkable improvement of the resistance to deformation under wear conditions which can reach a pressure of 3MPa and a sliding velocity of 1.5m/s.

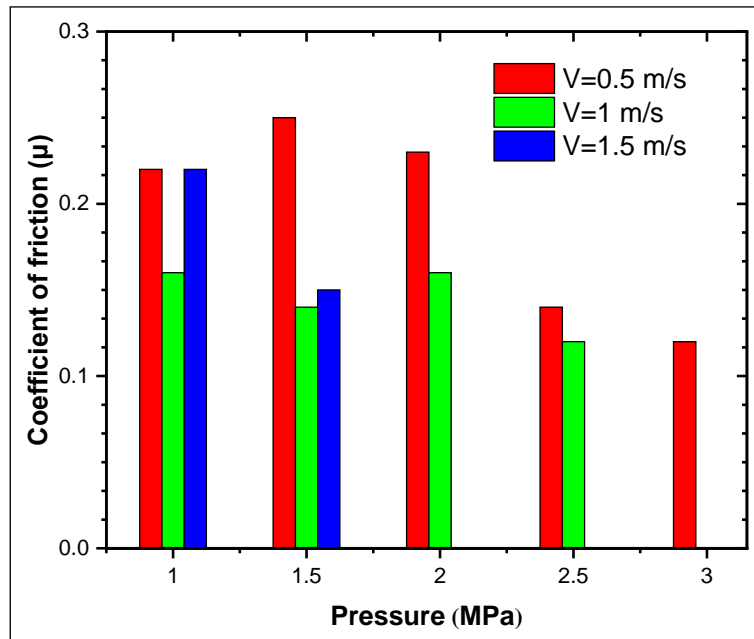
While the compositions Ep, Ep/Ca and Ep/Oil (Fig. 5 and Fig. 6) deteriorate at a load capacity limited to 1 MPa and 1 m/s at one hour of the time during the wear test, the Ep/Ca/Oil composition does not break or deform. As a result, the Ep/Ca/Oil is selected to evaluate its load capacity. Wear tests are carried out in a wide range of PV factor. The composite is tested at a nominal pressure in a range of 0.5 to 3 MPa and sliding speed of 0.5 to 1.5 m/s.

The tests are carried out with duration of one hour. The product PV (product of the normal pressure and the sliding velocity) increases considerably up to 3 Mpa.m/s where the pin starts to deform plastically as the temperature rises.

### 3.2.1 Effect of variation of speed and variation of pressure on coefficient of friction

Fig. 11 shows the evolution of the coefficient of friction of the composite at different sliding speeds and different pressure. For the Ep/Ca/Oil composition, the speed varies from 0.5 to 1.5m/s at a contact pressure going from 1 Mpa to 3 Mpa. One test is run for each composition.

The friction coefficient decreases with increasing pressure. Concerning the influence of velocity, the friction coefficient decreases at 1m/s but increases again at the speed of 1.5m/s, for this speed range, it can be seen that the friction coefficient gives a similar variation to the Stribeck curve [18], particularly for the pressures 1 Mpa and 1.5 MPa where we are able to measure at 1.5m/s.

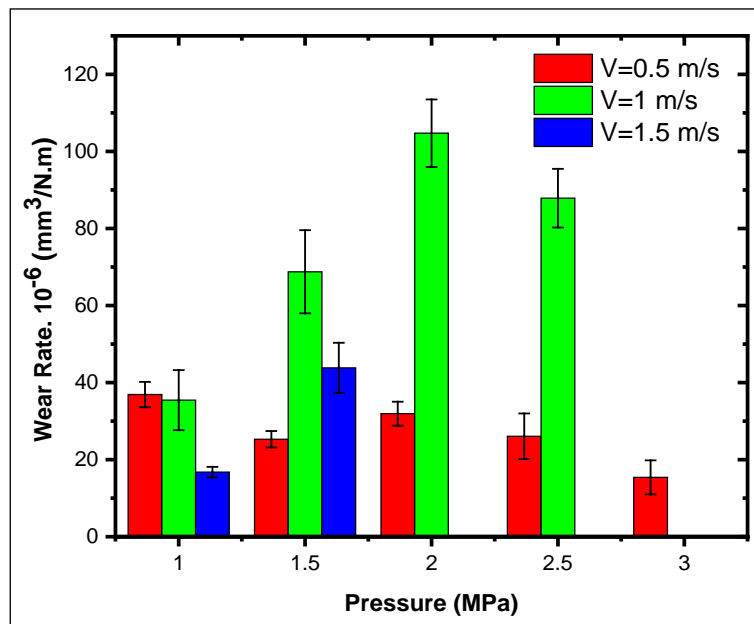


*Fig. 11 – Variation of speed and variation of pressure on the coefficient of friction*

### 3.2.2 Effect of variation of speed and variation of pressure on wear rate

Test pins are initially weighed on a single pan electrical analytical balance (Kern) that had a least count of  $10^{-8}$  kg. At the end of each test, specimens are cleaned and reweighed.

Various wear tests are used for testing wear property evolution under various sliding velocities and various pressures ranging from 1 to 3 MPa, and a sliding speed of 0.5 to 1.5 m/s. The wear rate shown in (Fig.12) is the average value of 3 tests during the wear process.



*Fig. 12 – Wear performance under different velocity and contact pressures*

According to the diagram in Fig. 12, the influence of sliding velocity on the wear rate shows that at 1 m/s, the wear rate has significant peaks compared to the speeds of 0.5 m/s and 1.5 m/s. For all 3 speeds, we can observe that the wear rate presents a diagram inversely proportional to the friction coefficient diagram. This leads us to say that the addition of



active carbon reduces the coefficient of friction but increases the wear rate, the oil film layer (Fig.9) reduces adhesive wear and decreases the coefficient of friction, which explains the resistance of the pin against deformation and failure under severe wear conditions.

For the different speeds used, we can see that the wear rate increases and reaches  $104.10^{-6}\text{mm}^3/\text{Nm}$  at 2 MPa and 1m/s, from this pressure it begins to decrease to  $15.10^{-6}\text{mm}^3/\text{Nm}$  at 3 MPa and 0.5 m/s.

## 4 Conclusion

In this paper, the tribological performance of lubricating oil-filled epoxy composites and activated carbon at various proportions and combinations are investigated under standard sliding conditions, ie, at 1 MPa and 1 m/s.

The addition of lubricating oil, by trapping, in the epoxy-based composites is made by using activated carbon; it can reach a concentration of 31w%. The results of the tests obtained at the end of this study show that the resistance to wear is improved, in particular at high contact pressure and at high sliding speeds.

The lowest wear rate is achieved for the epoxy loaded with 22w% of lubricating oil. The composition containing combined additives, that is to say 31w% of lubricating oil and 6w% of the activated carbon by mass, has a reduction in the coefficient of friction and allows to obtain a pressure of 3MPa and a sliding velocity of 1.5m/s. This means that the load capacity of the composite is improved by adding solid and liquid additives. Extensive chemical analyzes will certainly help to better understand the phenomenon of oil entrapment in the matrix.

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