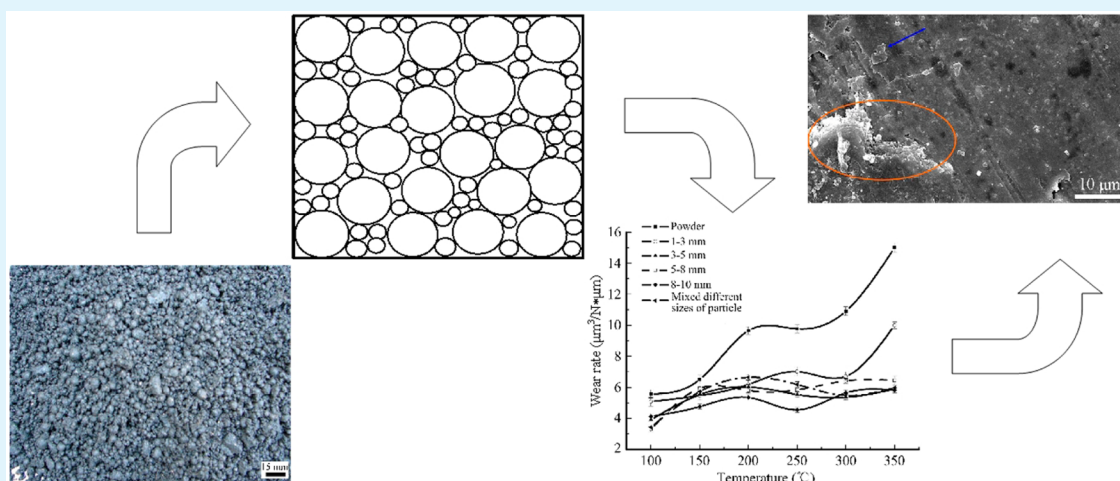


Evaluation of Wear Resistance of Friction Materials Prepared by Granulation

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ABSTRACT: The tribological properties of friction materials prepared by hot-pressing pellets of different sizes were experimentally investigated. Friction and wear tests of the specimens were performed and morphological analysis was carried out by investigating images acquired with both scanning electron and confocal laser microscopes. The highest friction coefficient of friction materials was obtained with pellets having 1–5 mm size. The lowest wear rate was obtained with pellets having 8–10 mm size. Specimens processed by mixing pellets of different sizes had the highest density and the lowest roughness and were the least expensive to fabricate. The results show that granulation generally enabled increasing the friction coefficient, decreasing the wear rate, and reducing the number of defects on the surface of friction materials.

KEYWORDS: granulation, friction materials, particle classification, friction coefficient, wear rate, wear resistance

1. INTRODUCTION

Research focused on obtaining materials with both high friction coefficient and low wear rate have been very intense in the last century.^{1,2} Such research studies have been motivated by the extensive use these friction materials have had in a very large variety of machineries including clutches, traction devices, brake systems,^{3,4} and other safety-critical components for transportation vehicles including trains, aircrafts, ships, and motor vehicles.

Several properties of these friction materials should be optimized to maximize wear resistance,⁵ especially when high mechanical loads are imposed and high increase of temperature is generated at the contacting surfaces.² While a known strategy to improve performance of breaking systems is to control their temperature,⁶ the selection of an appropriate chemical composition and microstructure⁷ still remain very critical for appropriately selecting the desired tribological properties of the friction materials.⁸ Having a uniform microstructure is

particularly important, for example, in the case of braking pads as their performance could otherwise drastically change during their progressive wear.⁹

Different manufacturing methods, including granulation,^{10–14} have been investigated to prepare friction materials with finely controlled microstructure. Granulation, which is the process used to agglomerate particles, finds applications in several different industries for the preparation of pharmaceuticals, fertilizers, and detergents and in the food processing industry.¹⁵ Granulation can decrease the segregation of the fibers, increase the porosity, decrease abrasion of mate surfaces, decrease the content of powders in the final product, and facilitate its transportation. However, no one has systematically reported

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about the effect of pellet particle sizes on the performance of friction material.

This article presents a thorough analysis aimed at quantifying the potential advantages of using agglomeration in the preparation of friction materials. In other words, the effect of pellets with different sizes on the tribological property of friction materials was systematically investigated. Specifically, section 2 presents the methods used to prepare the materials and the specimens investigated, and describe the procedures that were followed for carrying out both the friction and wear tests. Section 3 presents the results obtained for the wear and friction tests, investigates the morphology of the tested specimens, and discusses the outcomes of the performed analyses. Conclusions are drawn at the end of this article.

2. METHODOLOGY

2.1. Materials and Specimens Preparation. The friction materials consisted of 27 wt % compound mineral fiber (Beijing Hengnian Company, China), 13 wt % modified phenolic resin (Jinan Shenquanhaiwosi Chemicals Limited Company, China), 5 wt % vermiculite foam, 11 wt % iron powder (0.2–0.4 mm), 20 wt % precipitated barium sulfate (0.04–0.07 mm), 6 wt % petroleum coke (0.3–0.8 mm), 8 wt % artificial graphite (0.3–0.8 mm), 4 wt % alumina, 3 wt % antimony sulfide (0.1–0.2 mm), 1 wt % friction powder, and 2 wt % hard zinc sulfate. Among these raw materials, compound mineral fibers were selected as reinforcing ingredients. Modified phenolic resins were used as the binder. Alumina and iron powder were used as the abrasive. Antimony sulfide, petroleum coke, and artificial graphite were used as the lubricants. Friction powder, vermiculite foam, precipitated barium sulfate, and hard zinc sulfate were used as the particulate fillers and functional regulators.

Mixing and granulating equipment (JF805R, Changchun, China) was used to prepare the pellets. The raw material powder was first mixed on the mixer and subsequently poured into the granulator. The water content was fixed at 40% of the total weight of raw material powder, the stirring time was 15 min, and the speeds of the propeller and the drum were respectively 384 and 120 rpm, respectively. Figure 1 shows a specimen of the obtained pellets.



Figure 1. Specimens of the prepared pellets.

A grading sieve was used to sort the agglomerates in different groups according to their size. For each group, the agglomerates and other raw materials were mixed carefully using a blender (JF805R, Changchun, China) for 10 min. Then the mixed materials were pressed by compression molder equipment (JFY50, Changchun, China) for 30 min at a temperature of 160 °C and pressure of 40 MPa. To ensure resin completely cured, eliminate the residual stress, and remove a spot of residual volatiles, the mixed materials were dealt with heat treatment after hot pressing. The heat treatment was

subsequently imposed (as shown in Figure 2): the temperature was at 140 °C for 1 h in the first phase, 160 °C for 3 h in the second phase,

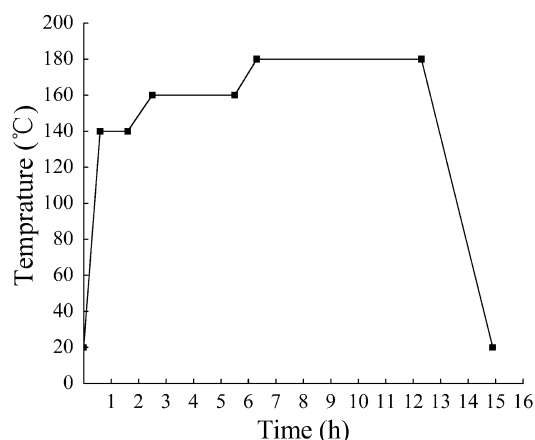


Figure 2. Heat treatment process used for the preparation of the testing specimens.

and 180 °C for 6 h in the third phase, respectively. Six specimens with standard size of 25 × 25 × 6 mm³ were prepared for the friction and wear tests.

Table 1 categorizes the obtained specimens into six groups. Group 1 includes specimens obtained directly from powder. Groups 2–5

Table 1. Group of Specimens

group	particle size
1	powder
2	1–3 mm (granulation)
3	3–5 mm (granulation)
4	5–8 mm (granulation)
5	8–10 mm (granulation)
6	mixed different sizes of particle (granulation)

include specimens obtained from agglomeration of pellets having the same size. Group 6 includes specimens obtained by mixing pellets of different sizes.

2.2. Procedures for Friction and Wear Tests. A friction test machine (JF150D-II, Changchun, China) was used for friction and wear tests. The material of the machine's disk was HT250, which had pearlite structure; the hardness of the disk was HB180–220; and the speed of the disk was 480 rpm and the press force was 0.98 MPa. Before the experiments were started, specimens underwent friction tests at room temperature (20 °C) for at least 10 min to ensure that the contact area between the friction disc and specimen was greater than 95% of the total area of the specimen.

Two sets of experiments, the fade and the recovery tests, were performed. For the fade tests, at least three specimens of each group listed in Table 1 were tested at six subsequently increasing constant temperatures, namely, at 100, 150, 200, 250, 300, and 350 °C. For each temperature, the fade test was stopped after 5000 revolutions (r) of the disk. After each test, the specimens were weighted with a scale having 1 mg accuracy and their thickness was measured in five locations with a micrometer having 1 mm accuracy.

For the recovery tests, the previous specimens were tested at five subsequently decreased constant temperatures, which is at 300, 250, 200, 150, and 100 °C. For each temperature, the recovery test was stopped after 1500 revolutions (r) of the disk.

The wear rate was calculated by using the following equation:

$$\Delta V = \frac{\Delta V}{SF} = \frac{A(h_1 - h_2)}{2\pi RnF} \quad (1)$$

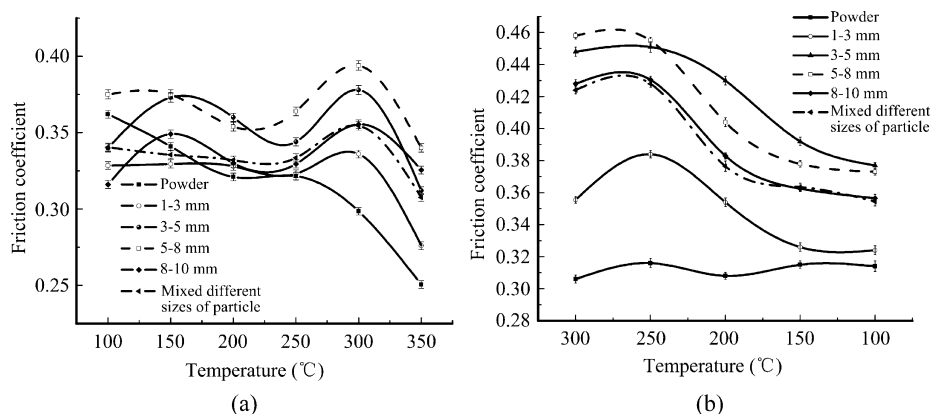


Figure 3. Influence of temperature on friction coefficient: (a) fade tests and (b) recovery tests.

where ΔW is the wear rate, A is the specimen size, ΔV is the worn volume, h_1 and h_2 are the thicknesses of the specimens before and after the tests, respectively, F is the average friction force, n is the number of revolutions of the friction machine's disk, S is the worn path, and R is the distance between the center of the specimen and the axis of symmetry of the spindle (in this study, $R = 15$ cm).

3. RESULTS AND ANALYSIS

3.1. Wear Properties of Friction Materials. Results of the fade and recovery tests are summarized in Figure 3. Figure 3a shows that the specimens prepared directly from the powder (group 1 in Table 1) degraded when temperature increased during the fade test. The shear strength and the friction coefficient (f) significantly decreased especially for temperatures higher than 250 °C. This decrease in performance was attributed to the thermal decomposition of the organic compounds (e.g., phenolic resins and fibers) with temperature, which was responsible for weakening the fiber–matrix bond. This is consistent with our previous research results.¹⁹

Interestingly, the behavior of the other groups was quite different. Specifically, the specimens having homogeneous size of the agglomerates (groups 2–5) reported an increase of f between 100 and 150 °C. The friction coefficient dropped from 150 and 200 °C and subsequently increased from 200 to 300 °C. A drastic decrease of performance was recorded when the temperature was further increased (see Figure 3a).

The improved behavior of the specimens obtained after granulation with respect to those obtained directly from the powder (group 1) was attributed to an improved resistance of the resins to high temperatures, as further investigated and described in the following sections of this manuscript. Specimens obtained by mixing both powder and agglomerates of different sizes (group 6, see Table 1) had hybrid behavior. In fact, f slightly decreased from 100 to 250 °C, and then increased between 250 and 300 °C.

Figure 3b shows that the f of the specimens prepared through granulation decreased with the decrease of temperature during the recovery tests. It should be noted that f for these specimens was however always higher than that for specimens prepared from the powder.

A relevant conclusion from the results presented in Figure 3 is that granulation generally improved friction properties especially at high temperatures. Specifically, the experimental results reported an average increase of f equal to 17.85–23.9% for group 1. Such an improvement was particularly significant when specimens had particles with size range of either 3–5 mm or 5–8 mm.

Figure 4 shows the relationship between ΔW , computed by using (1), and temperature. As is evident, the wear rates are

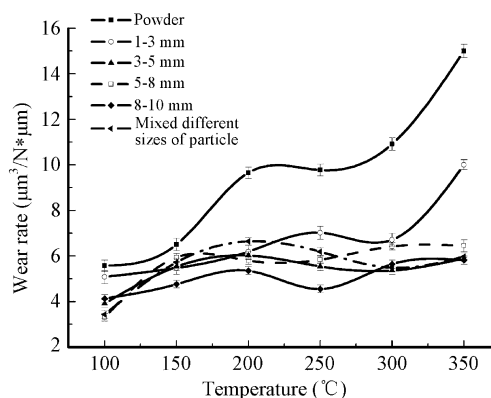


Figure 4. Wear rate of friction materials.

significantly influenced by the disc temperature. With the rising of test temperature, the wear rates for all specimens increase. This trend is in agreement with the earlier published study of Öztürk al.³ The wear rate for specimens obtained directly from powder was noticeably higher than that for the other specimens, especially for high temperatures. Specifically, granulation yielded a decrease in wear rate between 8.62% and 61.27%. Specimens with 8–10 mm particles had the best performance in terms of lowest wear rate, followed by specimens with 3–5 mm particles. It should however be noted that variation in wear rate among the groups 2–6 was negligible. Interestingly, ΔW of the specimens with the smallest particles (group 2) drastically increased for temperatures above 300 °C.

Improvements in wear resistance and friction associated with granulation could be attributed to a number of different factors including formation stage, size, and distribution of particles. In fact, although granulation does not alter the composition of friction materials, it however modifies the physical structure of the specimens. It should be noted that the strength of a structure depends on the size and distribution of its particles.¹⁶ The literature in fact reports that, for instance, wet stirring technology improves wear resistance as it forms a hard shell, consisting of a high-density water/powder layer, on the particles' surface.^{17,18}

In summary, results shown in Figures 3 and 4 indicated that specimens with particles having a medium size (e.g., 5–8 mm)

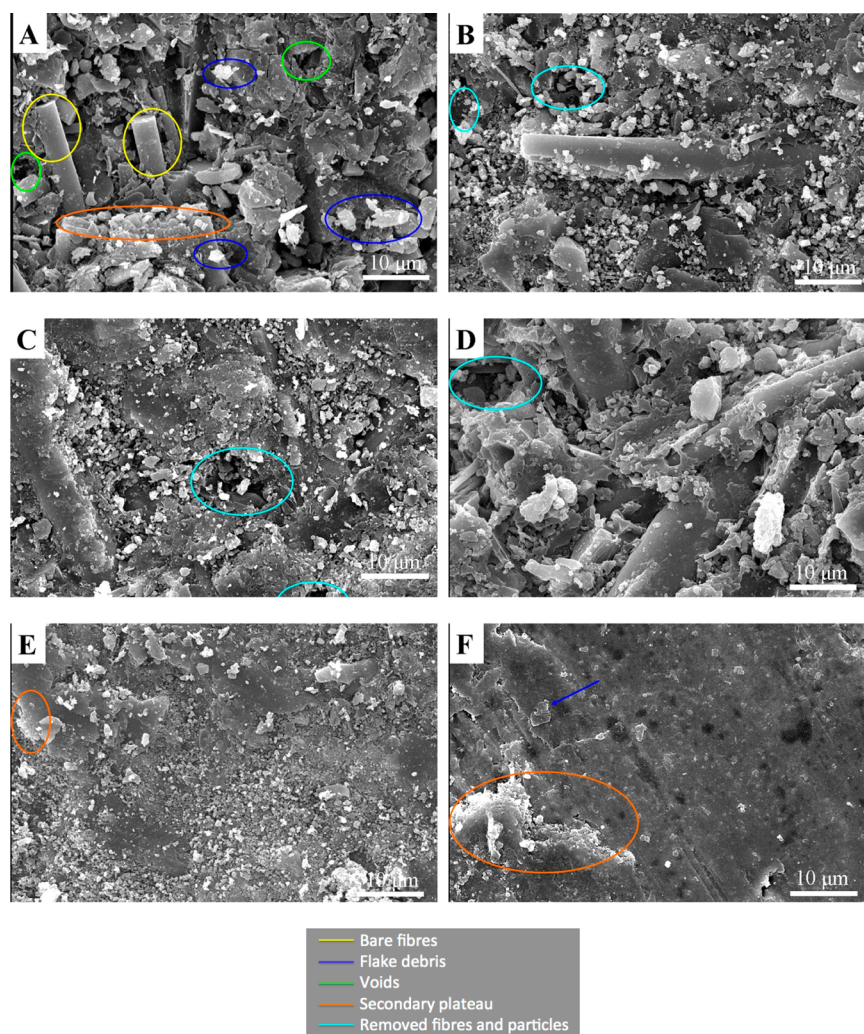


Figure 5. SEM images of specimens after tests. (A) Specimen of group 1 (from powder); (B) specimen of group 2 (1–3 mm particles); (C) specimen of group 3 (3–5 mm particles); (D) specimen of group 4 (5–8 mm particles); (E) specimen of group 5 (8–10 mm particles); and (F) specimen of group 6 (different size particles).

provided suitable compromise between high friction coefficient and low wear rate. In fact, particles with very small size, despite having a high ratio of surface over volume and a high interparticle bonding strength due to the high contact surface area between the particles, had a very uneven composition due to their size, which compromised the macroscopic wear properties of the specimens. On the other hand, particles with very large size, which had a very homogeneous composition, had an inherently limited contact surface area between particles and therefore a small interparticle bonding strength.

3.2. Analysis of Worn Surfaces. The analysis of plateaux, microcavities, wear debris, and roughness can provide useful information for investigating the tribological properties of friction materials.^{19–23} As proposed in the study of Li and Zhou, the surface morphology analysis of the tested specimens was therefore performed to study the mechanisms that governed wear during the performed tests.²⁴

Figure 5 shows six scanning electron microscope (SEM) images of the specimens that were tested. Specifically, Figure 5A shows a specimen of group 1. The surface appears to be very rough and the elements of the composite are not well-blended together. In fact, clear separations can be observed at

the interface between fillers and the phenolic resin. Large flake debris (blue circles in Figure 5A) detached from the surface and probably constituted the main cause of adhesive wear. The micrograph A provides evidence of voids (green circles in Figure 5A) and bare fibers (yellow circles in Figure 5A), which were responsible for high wear of this composite. Secondary plateaux were also presented (orange circles in Figure 5A). These plateaux consisted of organic material, such as charred, degraded, and softened resin and/or graphite, which were not bonded well to the composite.

Micrographs B, C, and D of Figure 5 refer to specimens of groups 2, 3, and 4 (see Table 1), respectively. Very heterogeneous surfaces with relative rough topography were presented. These SEM images show that the surfaces were damaged—there are in fact clear indications that elements of the composite, such as fibers and particles, were removed (see cyan circles in Figure 5). A small amount of wear debris was presented.

Figure 5E shows an SEM image of a specimen obtained through granulation from the largest pellets (8–10 mm) considered in this work. The surface, in this case, was quite smooth and there were few secondary plateaux (see orange circles in Figure 5), although the surface was covered with

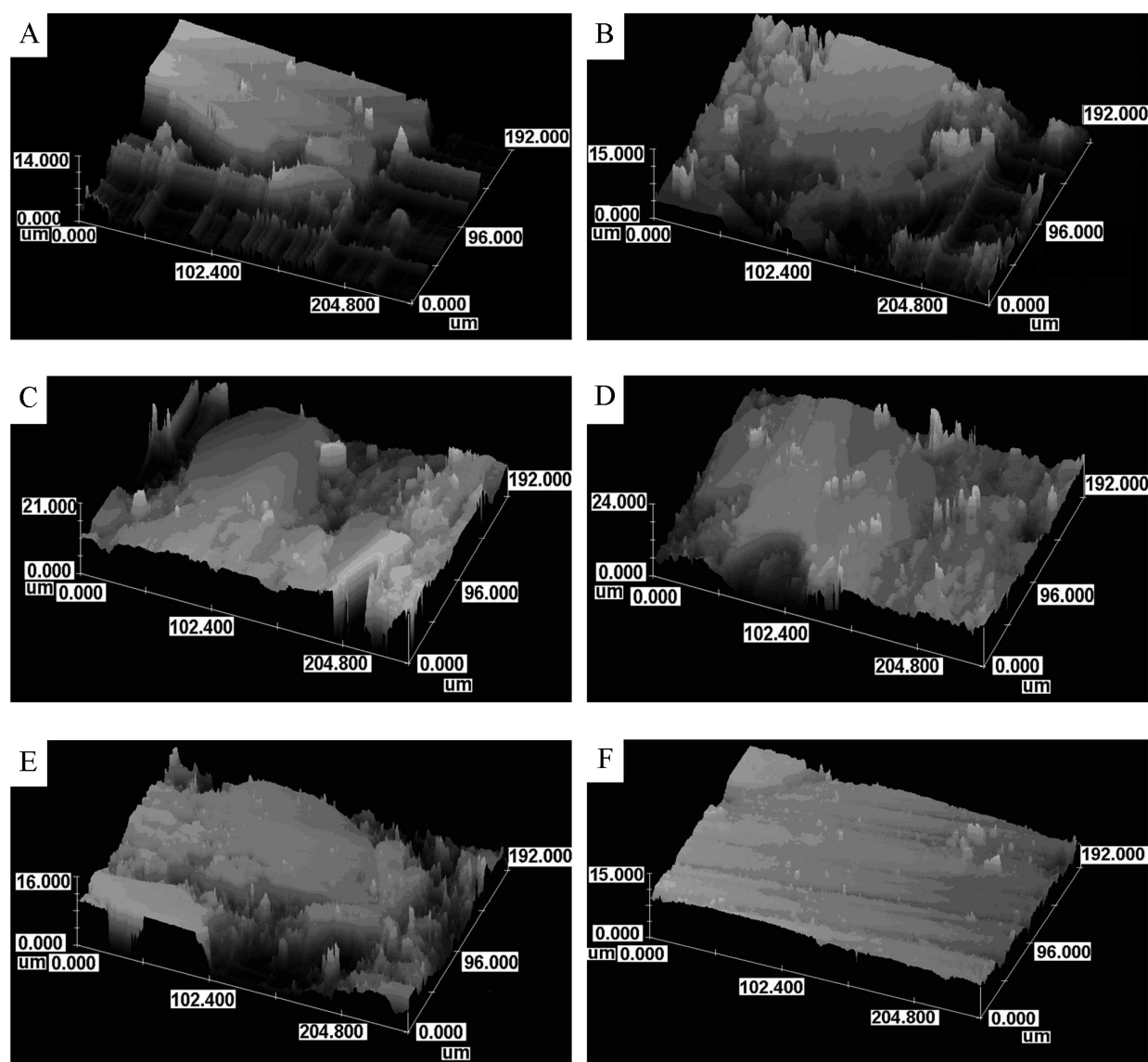


Figure 6. Three-dimensional reconstruction of the surface geometry of the tested specimens obtained through a confocal laser microscope. (A) Specimen of group 1 (from powder); (B) specimen of group 2 (1–3 mm particles); (C) specimen of group 3 (3–5 mm particles); (D) specimen of group 4 (5–8 mm particles); (E) specimen of group 5 (8–10 mm particles); and (F) specimen of group 6 (different size particles).

primary plateaux full of wear debris of various compositions. The different elements of the composite were well-blended together and little debris could be observed on the surface of the specimen. The moderately damaged surface reflects that specimens of group 5 yielded the highest wear resistance behavior (see Figure 4).

Figure 5F shows the micrograph of a specimen of group 6 (see Table 1). A large amount of medium-size debris (see blue arrows in Figure 5F) and thick secondary plateaux (see orange circles in Figure 5) were presented on the surface. Compared to the previous monographs, Figure 5F shows a very smooth surface with almost no bare fibers and separations at the interface between fillers and the phenolic resin. In an earlier study, Kumar et al.²⁵ also reported that the formation of secondary contact patches could cause the generation of friction film in the worn surface, and then those smooth friction films contributed to stable friction coefficient and lower worn rates.

An accurate analysis of the surface roughness of the specimens was performed by using a confocal laser microscope (OSL3000), which enabled obtaining a three-dimensional reconstruction of the surface geometry of specimens. It should be noted that roughness is an important parameter governing both friction properties and wear resistance.²⁶ Figure 6 reports six images associated with specimens of the six groups presented in Table 1.

The average (S_a) and the root-mean-square (S_q) roughness were computed over a surface area of $256 \times 192 \text{ m}^2$ for all the six investigated groups. Table 2 reports the computed values of roughness. It can be seen that both roughness and density decreased substantially when the size of the particles increased (groups 2–5). The specimens obtained without granulation (group 1) showed the highest density while their roughness was moderate. Interestingly, specimens of group 6 had very high density and the lowest roughness.

3.3. Discussion. Results showed that granulation generally improved tribological performance of friction materials. In fact,

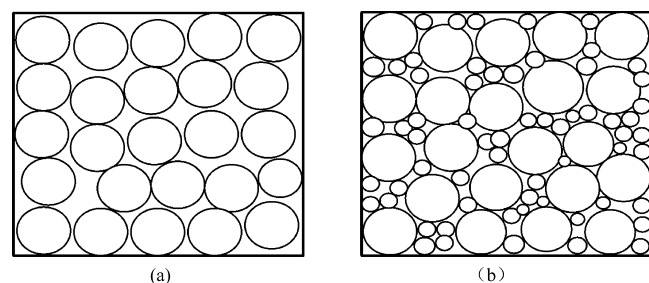
Table 2. Average Roughness (S_a), Root-Mean-Square Roughness (S_q), and Density of the Tested Specimens

group	specimens particle size	S_a (μm)	S_q (μm)	density (g/cm^3)
1	powder	1.252 ± 0.015	1.623	2.027 ± 0.13
2	1–3 mm (granulation)	1.679 ± 0.24	1.800	1.9887 ± 0.21
3	3–5 mm (granulation)	1.337 ± 0.018	1.640	1.9445 ± 0.32
4	5–8 mm (granulation)	1.346 ± 0.015	1.870	1.8875 ± 0.15
5	8–10 mm (granulation)	0.701 ± 0.011	0.921	1.8713 ± 0.18
6	mixed different sizes of particles (granulation)	0.478 ± 0.009	0.689	1.9967 ± 0.20

the value of the friction coefficient of the granulated specimens (groups 2–6) was relatively stable and was higher with respect to the specimens of group 1. In fact, f slightly fluctuated between 0.384 and 0.458 for temperatures that did not exceed 300 °C during the fade tests (see Figure 3). For the granulated specimens, the friction coefficient generally recovered its nominal value at the end of the recovery test. Granulation also caused a decrease of the wear rate as shown in Figure 4. Granulated specimens also did not appreciably increase their wear rate when the temperature was increased.

The performed topographic analysis showed that granulated specimens had less secondary plateaux, microcavities, wear debris, and roughness than that of the specimens obtained directly from powder (group 1). This result was in agreement with the lower wear rate recorded during tests performed with the granulated specimens. The improvement of tribological properties following granulation was associated with the fact that each pellet could be considered as a relatively independent unit with hard shell, which was strongly bonded to the other pellets of the composite.

A characteristic feature of granulation, as reported in Table 2, was the decreased density of the specimens. In fact, the gaps between pellets increased when the pellets' size increased. Figure 7a shows a schematic representation of a specimen with

**Figure 7.** Schematic diagram: (a) specimen with pellets having similar sizes; (b) specimen with pellets having different sizes.

large pellets. It can be seen that a large amount of space was not filled in the friction material. This fact might be considered a downside for either industries producing friction pads for vehicles, which would like to increase the life of their products, or for industries, such as the packaging industry, which are keen to reduce volume to reduce costs associated with storage and transportation. Improvement in density can be achieved by mixing pellets with different sizes as schematically shown in Figure 7.

Specimens of group 6 may therefore represent an optimal solution for many applications as they had high density, an overall high friction coefficient over a wide range of temperatures, and a low wear rate. The use of mixed size particles would also potentially reduce the cost of production by eliminating the sorting process otherwise required to sort pellets of different sizes.

4. CONCLUSIONS

The performed investigation provided evidence that granulation generally improves the tribological properties of friction materials. In fact, the friction coefficient and the wear rate of the specimens obtained from pellets were respectively 17.85–23.9% higher and 8.62–61.27% lower than that of the specimens prepared from powder, respectively. A reduced number of plateaux, voids, flake debris, and bare fibers were observed on the surface of the specimens obtained through granulation after the wear tests. Specimens with pellets in the 1–3 mm range had the highest friction coefficient during the fade tests. Specimens with pellets in the 3–5 mm range had the highest friction coefficient during the recovery tests. The wear rate had similar values for all specimens obtained through granulation, independently of the size of the pellets. The specimens prepared by mixing pellets having different sizes had the highest density and the smallest roughness after testing. The friction coefficient and wear resistance of these specimens were similar to the specimens that had pellets of uniform size. The performed analysis therefore yielded that specimens obtained by mixing pellets of different sizes could represent an optimal solution to obtain specimens with high friction coefficient, low wear rate, high density, low roughness, and low production cost (the use of pellets having different diameters does not require sorting the pellets by size).

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Notes

The authors declare no competing financial interest.

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