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Wear behavior of a new composite formulation, with TEOS addition, for abrasive vitrified grinding wheels

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ABSTRACT

Vitrified grinding wheels are used in many manufacturing industries to shape and finish metals and other materials in an efficient way. This work addresses a new approach with the use of tetraethyl orthosilicate solution (TEOS solution or silica sol), as an additive, in vitrified abrasive composites for grinding wheels. Different types of composite were produced using always the same materials and processing methods, but with different TEOS contents. Compared with a reference abrasive grinding wheel, the addition of TEOS promoted an improvement in the abrasive tool performance. The new formulation for vitrified abrasive composites showed better dimensional stability during processing (drying and sintering), and could be applied to the production of grinding tools with high dimensional accuracy. With the addition of 3.75 wt% of TEOS there was an increase in the strength of the matrix bonds. The porosity of the composite increased by 7% (from 43.6 to 46.9%), also improving its wear properties, with the quadrupling of the grinding ratio of the abrasive composite. In addition, the surface roughness of the composite decreased (from $R_a = 22.9$ to 8.4 µm) allowing to improve the surface finish of the workpieces.

1. Introduction

Grinding wheels are largely used in the manufacturing industry to shape and finish metals and other materials in an efficient way. They are essential in mechanical production whenever high surface quality is required, with low roughness and low residual stresses in the surface layer, and good dimensional accuracy even in complex shapes. Many industries such as aerospace, automotive, industrial heavy equipment, biomedical, to name only a few, depend on the grinding precision to produce more and more challenging geometric design parameters and surface finish requirements [1-7].

Among the wide variety of abrasive wheels that exist, the most used in all precision grinding applications are the vitrified abrasive wheels [8, 9]. Vitrified abrasive products have three main components, abrasive grains, the bonding material (or matrix) and the porosity; all playing an important role in the properties and performance of abrasive tools [10]. While the bonding material holds the abrasive grains together to form a

compact and stiff wheel, the abrasive grain is the cutting tool portion of the wheel. In turn, the porosity of the tool acts as a cooling agent, being a path for refrigerant/cutting fluids and sometimes a reservoir for cutting chips collection [6,11–15].

In a grinding wheel, the type of bonding material and its content influence the characteristics of the bonding bridges between the abrasive grains and, accordingly, the characteristics of the abrasive tool itself [8,16,17]. When the tool is in use, the nature of the fracture of the bonds and, consequently, the wear behavior of the grinding wheel/workpiece pair, depends on the vitrified matrix of the composite [3,9,18].

The bonding bridges between the grains of this type of composite must have sufficient mechanical strength to hold the grains, and at the same time they must break and release the grains, when they no longer perform their cutting role, allowing the cutting edges to be renewed [15, 17]. On the other hand, the glassy matrix should be as compact as possible in order to maximize the porosity, allowing for greater tool cooling/lubrication, as well as smoothing the pore walls and thus

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improving fluid flow [19]. Furthermore, during grinding operation, to ensure greater tool accuracy, the bonding material must have high dimensional stability with increasing temperature [20,21].

One way to reach these requirements could be the use of an organic source as an oxide-generating additive in the matrix. Tetra-Ethyl-Ortho-Silicate (TEOS) is a chemical compound whose formula is $(Si(OC_2H_5)_4)$, which will locally give rise to Si–O bonds after degradation. Generally, it is used in the sol-gel powder synthesis, for the crosslinking of silicones in the semiconductor industry, as a precursor of silica (SiO_2) within the framework of a sol-gel reaction. The remarkable property of TEOS is actually the formation of silica (SiO_2) by simple hydrolysis reactions releasing ethanol CH₃CH₂OH as described globally in Eq. (1) [22–24]. Considering the evolution of the partial reactions, the hydrolysis reaction (Eq. (2)) and the condensation reactions (Eqs. (3) and (4)), pure SiO₂ powders/glasses could be produced near room temperature.

$$Si (OC_2H_5)_4 + 2 H_2O \rightarrow SiO_2 + 4 CH_3CH_2OH$$

$$\tag{1}$$

$$Si (OC_2H_5)_4 + H2O \rightarrow Si (OC_2H_5)_3 - OH + CH_3CH_2OH$$
(2)

$$Si (OC_2H_5)_4 - OH + OH - Si (OC_2H_5)_3 \rightarrow (OC_2H_5)_3 \quad Si - O - Si \quad (OC_2H_5)_5 + H_2O$$

$$Si (OC_2H_5)_3 - OH + Si(OC_2H_5)_4 \rightarrow (OC_2H_5)_3$$
 $Si - O - Si (OC_2H_5)_3 + CH_3CH_2OH$

Commonly, the silica sol or TEOS solution is prepared using TEOS and deionized water as precursors, ethanol as co-solvent and nitric acid as catalyst [22–24]. The use of a sol-gel system has been investigated for many different applications [25]. Among them, in the field of abrasive grinding wheel manufacturing, the sol-gel method has been mostly investigated for the synthesis of abrasive grains [5], abrasive grains coating [26,27] or even for porosity inducers [28].

The use of the sol-gel method for the production of composite matrix is also presented in the literature. For example, Sibo Shen et al. produced a non-abrasive material - a new composite type of mullite fiber sealing pad - and found out that silica sol increased the material bulk density, from 0.31 to 0.40 g/cm³, whereas Young modulus increased from 0.97 to 3.24 MPa [22]. The literature that discusses the use of the sol-gel method for producing the vitrified matrix for abrasive grinding wheels is poor. However, lately, Wei Zhang et al. compared two kinds of vitreous bonds for diamond grinding tools, with the same chemical composition (Na₂O-Al₂O₃-B₂O₃-SiO₂), and the same sintering temperature; one type prepared by the traditional melting method and the other obtained by sol-gel method [29]. The sol-gel vitreous bonds presented better properties, as they form a denser network structure with a high bending strength of 137 MPa, 31.73% higher than the vitreous bond obtained by conventional melting method (104 MPa). Furthermore, the sol-gel method allowed a bond with lower coefficient of thermal expansion (CTE) than the vitrified bond obtained by the usual melting method. When increasing the sintering temperature, the CTE of the vitrified bond, prepared by the sol-gel method, first increased and then decreased, reaching a maximum value of 5.75 \times 10^{-6} $^{\circ}C$ $^{-1}$ at 720 °C, which was even lower than the minimum value for the vitrified bond prepared by melting method (7.02 \times 10⁻⁶ $^{\circ}$ C ⁻¹). It was also found that, among the vitrified diamond composites produced with the two studied vitrified bonds, those prepared by sol-gel method exhibit higher porosity and lower sintering shrinkage (sintered at 770 °C: 27.63% and 3.02%, respectively) than those prepared by melting method (sintered at 770 °C: 24.25% and 6.51%, respectively). This is a good feature, especially for the production of grinding tools with high dimensional accuracy. Vitrified diamond composites prepared by sol-gel method also presented a 24.7% higher bending strength (106 MPa) compared with the vitrified composite produced with a typical bond obtained by

melting method (85 MPa) [29].

In the present work, a new approach of the use of TEOS in abrasive composites for grinding wheel tools is addressed. A TEOS solution is incorporated as an additive in the initial oxide mixture (alumina abrasive grains + vitreous binder precursor powders + TEOS solution), acting then in the vitreous matrix of the vitrified abrasive composites (obtained by the traditional melting method). The materials and methods needed for the preparation of the samples of this research work are explained in section 2.1. The TEOS solution acts as an additive, since the oxide powders are the main source for the vitreous matrix. Abrasive composites with the addition of different TEOS contents (1.00, 2.25 and 3.75%) were produced and compared with an additive-free reference abrasive composite. Since the TEOS additive was added dissolved in a solution, an abrasive composite was also prepared with the excipient part of the solution (with addition of a neutral solution without TEOS), to evaluate the effect of the addition of this fluid phase into the abrasive composite, without the presence of TEOS. The characterization methods to investigate the effect of TEOS additive on the abrasive composite properties are described in section 2.2. The main effects of TEOS were observed in the dimensional variation, porosity, wear behavior, surface roughness and final microstructure of the composite. In section 3, the results of these characterizations are presented and discussed, and finally, in section 4, the main conclusions of this work are outlined.

2. Materials and methods

2.1. Sample preparation

(3)

(4)

In the process of manufacturing vitrified abrasive tools, the abrasive grains are mixed with the precursor bonding materials, pressed inside a mold to obtain a green compact, which is then dried and sintered [30, 31]. This bonding material plays a very important role in the development of the mechanical strength of bonding bridges between abrasive grains, and in the fracture and wear nature of vitrified grinding wheels. Thus, the properties of the vitrified bonding material are important as they also influence its binding conditions [3,9,18].

To assess the effect of TEOS on the abrasive vitrified bond, 3 different types of samples were produced for this study: i) the additive-free type, prepared using the traditional method, and named reference (REF); ii) the TEOS-added type of samples produced with 1.00, 2.00 and 3.75 wt % TEOS dissolved in a solution (weight percentage drawn over the abrasive mixture weight) – the corresponding type of samples was named according to the content of TEOS: T1.00, T2.25 and T3.75; iii) a similar solution, but without the TEOS reagent (neutral solution), was prepared and added to the abrasive composite instead of the TEOS solution in order to understand the effect of the liquid phase of the TEOS solution added on the abrasive composite. This abrasive composite prepared with the addition of the neutral solution was named N3.75 (liquid solution weight percentage equivalent to the T3.75 sample).

Apart from solutions addition (TEOS or neutral solution), the processing of all samples was always carried out in the same way as for the REF sample. First, the alumina abrasive grains and the vitrified bond precursor powder were mixed up, always using the same composition (Table 1). For samples with TEOS solution addition or with neutral solution addition, at the end of this mixture step, the respective solution

Table 1

Composition and sintering conditions for abrasive composite samples.

		REF
Vitrified bond precursor	Alumina (wt. %)	8.7
	Silica (wt. %)	56.6
	Fluxes (wt. %)	34.7
Abrasive grain fraction (wt. %)		88
Bond precursor fraction (wt. %)		12
Sintering temperature (°C)		950
Time at sintering temperature (h)		3

was added to the mixture, which was stirred again. Then, the homogeneous mixture was shaped by die-pressing, with 27.5 MPa of compaction pressure. Then, the obtained green compacts were dried in an oven (Venticell) at 100 °C. After drying, the compacts were put into a kiln and sintered according to the processing conditions presented in Table 1.

2.1.1. Preparation of solutions

To prepare the TEOS solution, in addition to the TEOS reagent, three other chemical reagents were used, namely: water (H₂O), nitric acid (HNO₃) and ethanol (C₂H₆O). In this solution, TEOS is the source of silica since water hydrolyses TEOS so that it can polymerize. Ethanol is a co-solvent that is miscible with both TEOS and water helping to get both into the same phase so they can react. Nitric acid is a catalyst that helps hydrolysis to happen more quickly. The neutral solution was prepared in the same way but without adding TEOS. Table 2 shows the composition of TEOS and neutral solutions.

Since, to form SiO₂ from the TEOS solution, the limiting chemical element is Si and taking into account that the remaining components are volatile and will evaporate during the drying and firing of the composite, it is possible to estimate the addition of SiO₂ generated in the abrasive composite from the added amount of TEOS. Table 3 shows the content of SiO₂ that is kept in the abrasive composite after sintering, based on the added TEOS content.

For this work, disc-shaped samples (\emptyset 62.35 \times 8.00 mm) were produced with a central hole (\emptyset 8.11 mm), which were used for wear tests, dimensional variation tests, surface roughness measurements and scanning electron microscopy (SEM) analysis.

2.2. Sample characterization

2.2.1. Dimensional variation

During the processing of abrasive grinding wheels or even during the use of these tools, due to temperature changes, unwanted dimensional variations can occur [28,29]. Dimensional variations of grinding wheels during processing require one more step of resizing in order to obtain a uniform geometry before using the tool. In this situation, raw material and time will be wasted unnecessarily. On the other hand, these dimensional variations, associated with thermal changes during the use of the tool against the workpiece, induce a lower tool precision and a less accurate operation [20,21,29].

The dimensional variations of the composites under study were evaluated by the difference in dimensions between the end of pressing (pressing-die dimensions) and the end of sintering (sample dimensions). Dimensional data were collected by measuring the pressing-die and the sintered samples, REF and T3.75, with an Absolute Digimatic caliper (Mitutoyo). These data were compared with the dimensions of the pressing-die used for compacting all the samples. The dimensional variation was also evaluated more specifically, during the drying stage of the composites processing. For this characterization, samples of green composites REF, N3.75 and T3.75, were prepared, all under the same conditions, equal mass and compaction pressure (0.35 g and 27.5 MPa, respectively), until the pressing step, with \emptyset 6 \times 5 mm. Dimensional data were collected using a Thermomechanical analyzer equipment -TMA (TMA Q400 - TA Instruments). To simulate the usual drying stage of the composite, the thermal cycle was performed in three steps: increase in temperature at a rate of 2 °C/min from 30 °C to 70 °C, stay at 70 °C for a thermal stage of 1 h and return to 30 °C maintaining the same

Table 2	
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Reagent	TEOS solution (wt.%)	Neutral solution (wt.%)
TEOS	18.41	-
H_2O	19.10	23.41
HNO ₃	1.45	1.77
C_2H_6O	61.04	74.82

Table 3

 SiO_2 content obtained from the addition of TEOS in the abrasive composite.

Composite nomenclature	Added TEOS (wt. %)	Remaining SiO ₂ (wt. %)
REF	0.00	0.00
T1.00	1.00	0.29
T2.25	2.25	0.65
T3.75	3.75	1.08

rate of 2 °C/min for temperature decrease. As support for the interpretation of the result curves, CTE of the three abrasive composites was determined in the first step of the thermal drying cycle. With this dimensional characterization, it was intended not only to understand the influence of the additive in a TEOS solution on the expansion/shrinkage of an abrasive composite during processing, but also on its accuracy as a tool (when used).

2.2.2. Porosity measurements

Porosity is one of the most important characteristics in a grinding wheel, as it positively influences its performance and defines the final surface quality of the workpieces. In a grinding wheel, the pores support the supply of coolants to the grinding zone and the removal of chips and residues [15]. Additionally, in systems with higher porosity, the bonding bridges become thinner (lower contact area) and the interaction between bond and workpiece is significantly reduced (lower friction), resulting in less thermal degradation or damage [14]. In order to understand the influence of the additive on the porosity of the abrasive composite, the open and closed porosities of the REF and T3.75 composites were determined. For this purpose, the composite samples prepared with \emptyset 36 \times 10 mm (dried at 105 °C) were weighted to obtain their dry weight (D). Then, the samples were immersed into boiling distillated water for 2 h in order to completely impregnate all open pores, remaining there while cooling for 24 h. The suspended weight (S) of each soaked sample was determined while it remained suspended in water. Afterwards, for the determination of the saturated weight (W), each sample was lightly blotted with a soft, moistened cotton cloth to remove all water droplets from its surface.

The apparent porosity or open porosity (P_A), expresses the ratio of the volume of open pores in the sample to its total volume and was calculated according to Eq. (5). In turn, based on the volumes of open pores (V_A , Eq. (6)), bulk material (V_{bulk} , Eq. (7)), and closed pores (V_C , Eq. (8)), the closed porosity (P_C), which is the ratio between the volume of closed pores in the sample and its total volume, was calculated according to Eq. (9).

$$P_A, \% = \left[\frac{W-D}{W-S}\right] \times 100 \tag{5}$$

$$V_A = \frac{W - D}{d_{H_2O}} \tag{6}$$

$$V_{bulk} = \frac{D}{d_{bulk}} \tag{7}$$

$$V_C = \left(\frac{D-S}{d_{H_2O}}\right) - V_{bulk} \tag{8}$$

$$P_C, \% = \left[\frac{V_C}{V_{bulk} + V_A + V_C}\right] \times 100 \tag{9}$$

2.2.3. Wear tests

During a grinding operation, more than 90% of the dust particles come from the material being ground. However, the remaining part of the loosed material is related to the wear of the grinding tool and determines both the life of the abrasive part and the quality of the surface finish [3].

The best way to evaluate the tribological behavior of an abrasive

composite is to submit it to a wear test. Thus, for comparison purposes, all the prepared abrasive composites (REF, T1.00, T2.25, T3.75 and N3.75) were subjected to wear tests keeping the same conditions for all tests. For each type of abrasive composite, four disc-shaped samples \emptyset 62.35 \times 8.00 mm were tested. Wear tests were carried out in a pin-ondisc geometry, with distillated water as a lubricating/cooling fluid. All wear test conditions and evaluation methodology were designed to allow the measurement of the wear rate of both mating surfaces, by weight loss, expressed in grams per meter (g/m). Since under usual conditions the metallic material of the workpiece is transferred to the grinding wheel, not allowing the determination of the mass variation, alumina pins (Ø5 mm) were used as counterface (from CERATEC, 99% purity). The normal applied load, the sliding speed and the sliding distance were kept constant at 20 N (corresponding to a nominal contact pressure of 1.02 MPa), 0.5 m s⁻¹ and 1800 m, respectively. In addition, the determination of the grinding ratio (G ratio) was adapted from the traditional analysis of the volumetric wear relation between the workpiece and the grinding wheel. For each tribological pair, the G ratio was calculated by the relation between the values of the wear rate of the pin and opposing composite disc, according to Eq. (10).

Grinding ratio =
$$\frac{Pin \ wear \ rate \ (g/m)}{Disc \ wear \ rate \ (g/m)}$$
 (10)

The grinding ratio is a way of evaluating the performance of a grinding wheel, typically the higher the grinding ratio the more efficient the grinding wheel. Although these tests do not represent the actual conditions of use of these tools, they allow a prior comparison of a new formulation of an abrasive composite with a reference material (REF) already known and used (e.g. for surface grinding, saw sharpening and internal grinding). Sliding speed is lower than for usual conditions, but also the counterface (alumina) is much harder than usual, creating particularly difficult contact conditions. This adapted method of testing was already validated in a previous work [3], for studying the effectiveness of a grinding material by comparison with another material of proven effectiveness (already being used industrially). In fact, this evaluating methodology has even supported the development of different abrasive composites (mainly vitrified and resin-bonded abrasives).

2.2.4. Surface roughness measurements

Grinding is a surface finishing process for parts in which it is intended to achieve a high level of dimensional accuracy, low surface roughness and low residual stresses in the surface layer [4,6]. For this, the surface roughness of the abrasive tool itself is extremely important, as it will define the topography of the workpiece. Thus, prior to the wear tests, the surface roughness of the REF and T3.75 samples was characterized using a Surftest SJ-210 surface roughness measuring instrument. Surface roughness was measured in 3 different zones of each sample: outside the wear track (unworn area), perpendicular to the wear track (transverse direction inside the wear track) and tangent to the wear track (longitudinal direction inside the wear track). A 3 mm stylus travel was used on all tests. For each measurement, a surface profile was obtained and the arithmetic mean roughness, Ra (μ m), was determined.

With these measurements, comparing the surface roughness outside and inside the wear track, in each composite sample, it was intended to evaluate the finishing quality of the future grinding tool. Furthermore, to compare the two types of abrasive composites under study (REF and T3.75), the effect of the TEOS additive on the surface roughness of the composites was also evaluated. The results were analyzed, taking into account the influence of the surface topography of the tool on the surface topography of the workpiece.

2.2.5. Scanning electron microscopy analysis

To better understand, relate and interpret all the results obtained by the characterization techniques described above, it is important to know the microstructural changes due to wear tests that occur in composites with and without the TEOS additive. A detailed analysis of the surface morphology of the REF and T3.75 abrasive composites, inside and outside of the wear track, was performed using a scanning electron microscope system – NanoSEM - FEI Nova 200 (FEG/SEM).

3. Results and discussion

3.1. Dimensional variation

The dimensions of the sintered samples REF and T3.75 are presented in Table 4, as well as the respective dimensional variations since their removal from the pressing die. The values shown in Table 4 are an average obtained from several measurements, in all samples (three for each type of composite), of the outer diameter, the hole diameter and the thickness.

In Table 4, analyzing the dimensional variations from the pressing die to the end of sintering, in samples REF and T3.75, it can be seen that with the addition of TEOS solution (T3.75) the composite presents smaller dimensional changes when compared to the REF composite.

The three prepared cylindrical samples ($\emptyset 6 \times 5$ mm), made of composite REF, N3.75 and T3.75, were tested in a TMA equipment for comparison between them. During the TMA analysis, only the dimensional variation of the height of the cylinder (maintaining a constant contact with air, allowing evaporation) was evaluated. Fig. 1 shows the corresponding curves obtained in these tests.

On the first stage (heating) the sample expansion is similar for samples REF and T3.75 with CTE of 17.13 and 17.24 µm/(m.°C), respectively. Sample N3.75 has a higher CTE of 24.75 μ m/(m.°C), due to the extra liquid phase (without TEOS crosslinking effect), expanding freely, between the solid particles. During the isothermal stage at 70 °C, sample N3.75 shows a significant contraction, which indicates the moment of evaporation of the excess liquid phase (~55 min), composed mainly of ethanol and water. This effect is not present in the other two sample types. The composite with the addition of TEOS solution (T3.75) presents a different behavior: there is a slight decrease in the sample dimension, during the stage at 70 °C, showing that the formation of organosilicon/silanol is occurring simultaneously with the formation of ethanol from the hydrolysis reaction (cf. Eq. (2)), but by "freezing" the composite bonds and drastically reducing shrinkage due to ethanol evaporation. Among the three types of samples, only T3.75 recovers its initial dimension during the cooling step of the studied drying thermal cycle. The TMA results also show that the main contraction of the REF sample (Table 4) does not occur during the drying step, but during sintering. On the other hand, the addition of the TEOS solution seems to "freeze" the bonds and microstructure of the green composite, explaining its lower dimensional variation after sintering (Table 1).

3.2. Porosity measurements

Table 5 indicates for the REF and T3.75 abrasive composites, the open, closed and total porosity, which corresponds to the sum of the two previous values.

Samples T3.75, with the addition of a solution with 3.75% of TEOS, show a clear increase in total porosity compared to the reference, of about 3.17%. This increase is mainly due to the variation in open porosity. For the same production conditions, all samples produced with the addition of the TEOS solution showed higher porosity. The presence of the TEOS solution plays an important role in the vitreous matrix, increasing the porosity of the composite. All the reagents found in the TEOS solution (mainly ethanol), seem to occupy a certain volume during the pressing step, giving rise to well-distributed and organized spaces between the grains. During the drying step, a partially hydrolyzed siloxane/silanol is formed and, furthermore, the sintering leads to the formation of silica. This entire process is characterized by maintaining the initial position of the abrasive particles, first being fixed by the silica sol (drying), and then, in the sintering stage, by the silica, when volatile

Table 4

Dimensional variations of samples from pressing to the end of sintering (950 °C).

	Pressing die cavity dimensions (mm)	Dimensions of the sintered abrasive composite			
		REF (mm)	Variation (%)	T3.75 (mm)	Variation (%)
Outer diameter	62.35	62.53	0.29	62.35	0.00
Hole diameter	8.11	8.26	- 1.85	8.11	0.00
Thickness	-	8.14	- 1.75	7.97	- 0.38



Fig. 1. Dimensional changes of the abrasive composite during the drying thermal cycle: REF, N3.75, and T3.75.

 Table 5

 Open, closed, and total porosity of samples REF and T3.75.

Porosity	REF	T3.75
Open porosity	42.04	45.46
Closed porosity	1.56	1.32
Total porosity	43.61	46.78

compounds give rise to empty spaces, increasing the porosity of the abrasive composite. The production of vapor species as condensation reactions occurs (Eqs. (3) and (4)), and at the same time a reorganization of the vitreous bonding material over the grains [10,22] may also be at the origin of the increase in the porosity of the abrasive composite.

3.3. Wear tests

The different types of samples were tested to characterize the wear behavior (REF, T1.00, T2.25, T3.75 and N3.75). To analyze and compare the wear behavior of all types of samples, four samples of each type were tested. The friction coefficient was recorded during all wear tests, always showing similar behavior for all types of samples. For all tests, the steady-state friction coefficient assumed values between 0.18 and 0.20.

The results of wear tests are shown in Figs. 2 and 3. While Fig. 2 shows the wear behavior of the new composite, as a function of the amount of TEOS added (REF, T1.00, T2.25, and T3.75 composites), Fig. 3 compares samples REF, N3.75 and T3.75, to evaluate the physical effect of TEOS on the composite. Figs. 2a and 3a show the variation of the wear rate of the abrasive composites, Figs. 2b and 3b refers to the wear rate of the alumina pin (counterface) when against the different abrasive composites, and Figs. 2c and 3c show the grinding ratio for each tribological tested pair.

The results in Fig. 2 indicate that when adding a TEOS solution to the abrasive composite, the wear rate of the composite reduces by \sim 87%, while the wear rate of the pin reduces by \sim 50%. These changes in the wear behavior of the composite result in an increase in the grinding ratio.

The increase in grinding ratio with increasing TEOS content in the composite more than quadruples the grinding ratio for an addition of 3.75% TEOS compared to the reference material (0.00% TEOS). Despite being more porous, the composite with the addition of TEOS solution (T3.75) has greater wear resistance, with a lower wear rate than the composite without TEOS (REF). Thus, there is an increase in the wear resistance of the abrasive composite with the increase of its TEOS solution content.

In Fig. 3, composites REF, N3.75 and T3.75 are compared regarding their wear behavior. An improvement in wear resistance is observed for the composite with the addition of neutral solution (N3.75) so that, compared to the reference composite (REF), the grinding ratio value has doubled from 0.029 to 0.064. The same behavior was observed from the N3.75 composite to the T3.75 composite (grinding ratio doubled from 0.044 to 0.122), showing the effect of TEOS in the added solution, which clearly increases the wear resistance of the abrasive composite.

Knowing that the TEOS solution is in the liquid state (ethanol + TEOS + water + nitric acid), and also considering the equation of silica formation from TEOS and water (Eq. (1)), the combined effect of siloxane/silanol formation with the presence of a liquid phase during the pressing step of the composite could be the cause of these results.

First, as a liquid, the TEOS solution seems to act mechanically during the pressing step, moving within each space in the mixture. In this way, in addition to increasing the porosity of the composite, it distributes the compaction pressure in an isotropic way, ensuring greater compaction in the specific area of the grain and vitrified binder bridges [22]. On the other hand, the formation of silica (from the transformation of the organosilicon compound during sintering) can change the properties of the vitrified bond, making it more stiff, and justifying the increase in the wear resistance of the composite with the increase in the addition of the TEOS solution. Thus, there is an improvement in the mechanical properties of these vitrified connections, namely in the resistance to shear forces while using the tool.

3.4. Surface roughness measurements

As mentioned in section 2.2.4, surface roughness measurements were performed on the tested abrasive wheels in three different zones, namely outside the wear track, inside the wear track in the perpendicular direction and inside the wear track in the tangent direction.

The surface roughness results (Table 6), indicate that both inside and outside the wear track, the roughness of the sample is reduced with the addition of TEOS in the composite. Overall, a 63% reduction in surface roughness outside the wear track was observed for the abrasive composite. Inside the wear track a surface roughness decrease of \sim 18% also occurs when adding TEOS to the composite. Fig. 4 shows representative profiles of the topography of two composites where measurements were performed.

In Fig. 4, regardless of the location of the analysis, there is always a notable difference between the topography of the REF and T3.75 composites. Compared to normal abrasive (REF), the abrasive composite with the addition of the TEOS solution (T3.75) clearly presents a more regular and homogeneous topography, thus justifying the lower values of surface roughness for the T3.75 composite (Table 6). After the wear test, the average of the R_a values measured on the wear track obtained in the perpendicular direction and in the tangent direction resulted in relatively close values: 14.80 μ m for the REF sample and 12.06 μ m for



Fig. 2. Wear results of the abrasive composites: REF, T1.00, T2.25 and T3.75: (a) Wear rate of the abrasive composite disc. (b) Wear rate of the alumina pin counterface. (c) Grinding ratio of the tribological pair.

Fig. 3. Wear results of the abrasive composites: REF, N3.75 and T3.75: (a) Wear rate of the abrasive composite disc. (b) Wear rate of the alumina pin counterface. (c) Grinding ratio of the tribological pair.

Table 6

Surface roughness measurements (Ra, in μm) for REF and T3.75 samples after wear tests.

R _a measurement line	REF (µm)	T3.75 (μm)
Out of wear track	22.91 ± 0.59	$\textbf{8.42}\pm\textbf{0.30}$
Wear track (perpendicular)	13.95 ± 2.18	11.55 ± 2.85
Wear track (tangent)	15.64 ± 0.00	12.57 ± 0.32



Fig. 4. Profiles of the topography of the abrasive composites obtained out of the wear track, perpendicular to the wear track, and tangent to the wear track: (a) REF sample and (b) T3.75 sample.

the T3.75 sample. It is worth mentioning that, after the wear test, R_a increases in the case of the T3.75 composite from 8.42 to 12.06 µm (average value for measurements in perpendicular and tangent directions) and this phenomenon could be attributed to grain pull out as well as some matrix loss/silica phase loss. In the case of the REF abrasive composite, when in the green state, less packing/organization of the abrasive grains is expected, that is, the rearrangement of the grains is difficult, since without TEOS solution, the vitreous bond precursor is less fluid than in the T3.75 composite. Consequently, the surface roughness of the REF composite reaches high values, in the order of 22.90 µm (Table 6). Regarding the wear track, in both cases the surface degradation mechanisms identified were similar, resulting in grain pullout and matrix loss. In the REF sample, these mechanisms tend to be enhanced, occurring from poorly rearranged grains, so after the wear test, the surface roughness of the REF samples is higher than that of the

T3.75 composite (14.80 μ m versus 12.06 μ m, considering the average of the values obtained for each composite in the perpendicular and tangent direction presented in Table 6).

3.5. Scanning electron microscopy analysis

The surface morphological features of the REF and T3.75 samples were analyzed by scanning electron microscopy outside and inside the wear track. Fig. 5 shows the microstructural characteristics of the unworn surfaces of the normal abrasive composite REF (Fig. 5(a)) and of the abrasive composite with the addition of a solution with 3.75% of TEOS, T3.75 (Fig. 5(b)).

The morphological features shown in Fig. 5 agree with the obtained surface roughness results (Table 6 and Fig. 4), as the REF sample presents a rougher appearance (Fig. 5(a)) compared to the T3.75 sample (Fig. 5(b)). In TEOS solution-added samples (Fig. 5(b)), it seems that alumina grains are well organized, clearly showing that the addition of TEOS aids in the rearrangement of the abrasive grains and influences the way the vitrified bond covers them. The greater organization/rearrangement of the abrasive grains can be caused by a more homogeneous distribution of compaction pressure (due to the liquid phase during the pressing step). In addition, the vitrified matrix seems to become more fluid during sintering [22], as can be seen in Fig. 5(b)), due to the absence of vitreous bonding lumps, which are observed in Fig. 5(a) for REF composite (which are points of shear stress concentration during tool operation). With the addition of TEOS there is no apparent increase in terms of space between grains. The porosity calculation (Table 5) may indicate that the extra open porosity formed in the TEOS samples is mainly due to the new arrangement of the grains, but also to the new arrangement of the vitreous bonding material, on and between the grains, clearly exposing their cutting edges.

Fig. 6 shows the worn surface morphologies of the two abrasive composites, REF (Fig. 6(a)) and T3.75 (Fig. 6(b)).



Fig. 5. Scanning electron micrographs of the unworn surface of abrasive composites. (a) REF, (b) T3.75.





Fig. 6. Scanning electron micrographs showing the worn surface of abrasive composites. (a) REF, (b) T3.75.

The morphological features of the worn surfaces (Fig. 6) corroborate the obtained wear results, which were characterized by a significant decrease in the wear rate by the addition of TEOS solution in the abrasive composite (Figs. 2(a) and Fig. 3(a)). The worn surface of REF composite (Fig. 6(a)) denotes localized abrasive wear on the protruding abrasive grains, with the formation of some wear flats (or plateaus) on its contact surface, formed as a consequence of fine scale abrasive wear [32]. Another wear mechanism observed is the fracture of the vitrified bonding bridges and the consequent detachment of abrasive grains.

Although samples with the addition of TEOS solution have a denser and stiffer vitrified bond, the wear mechanisms are similar in both types of samples, REF and T3.75, as well as in all abrasive composites tested in this study. Thus, as in the case of the REF composite, for TEOS solutionadded samples (Fig. 6(b)) fine scale abrasion with formation of plateaus on protruding grains and detachment of abrasive grains were the dominant wear mechanisms observed. However, it can be observed on the worn surface of the sample with TEOS (Fig. 6(b)) a greater amount of small wear debris (compared to the size of the abrasive grains) generated by the wear of grains and binding material. Furthermore, as with the addition of TEOS the vitrified bond becomes more resilient, holding the grains for longer than in the composite without TEOS, the formation of wear plateaus is more significant (larger flat areas), which proves that the matrix maintains a high capacity to retain abrasive grains despite being subjected to wear.

4. Conclusions

In this work, a simple method for adding TEOS (tetraethyl orthosilicate solution) in abrasive composites to grinding tools, namely in solution, was developed. This method does not significantly interfere with the traditional grinding wheel manufacturing process and positively alters the properties of the vitrified matrix. Abrasive composite samples were produced, always using the same processing conditions, except for the additive content: 0.00, 1.00, 2.25, 3.75% TEOS (in wt.%).

Sintered reference samples (REF), without TEOS, and samples with 3.75% of TEOS (T3.75) were dimensionally compared showing that, for the same processing conditions, samples with TEOS are more faithful to the pressing die cavity dimensions. Tests performed in TMA equipment showed that, during the drying step, the composite with the addition of TEOS presents a higher dimensional stability, despite the higher amount of liquid phase added to the mixture.

Regarding porosity, the addition of the TEOS solution generated part of the vitrified bonding bridges of the abrasive composite, giving rise to greater porosity and smoothing the pore walls, allowing a greater cooling/lubrication of the tool, and improving the fluid flow.

Despite being more porous, composites with the addition of TEOS solution have higher wear resistance, presenting lower wear rates than the composite without TEOS. The wear test of another abrasive composite produced with the addition of a neutral solution (N3.75), compared with composite with TEOS solution addition (T3.75), showed that this increase in wear resistance is due to a combined effect: the formation of silica (from TEOS) and the presence of a liquid phase during the pressing step, which seem to increase the mechanical strength of the vitrified bond, improving its wear behavior.

SEM analyses and surface roughness results showed that TEOS solution-added abrasive composite (T3.75) clearly has a more regular and homogeneous topography ($R_a = 8.42 \mu m$) compared to the reference abrasive ($R_a = 22.91 \mu m$). In the wear track, both abrasive composites showed similar wear mechanisms: abrasion of the protruding grains, with the formation of wear flats (or plateaus), and breaking of the vitrified bond bridges, leading to the detachment of abrasive grains. However, with the addition of TEOS the vitrified bond becomes more resilient, holding the grains for longer than in the composite without TEOS. Consequently, in this case the formation of wear plateaus is more significant (larger flat areas), proving that the matrix maintains a high capacity to retain abrasive grains despite being subjected to wear.

The methodology proposed in this study for a new composite formulation, with the addition of TEOS for vitrified grinding wheels, strengthens the vitrified bridges between grains, improving the mechanical and wear properties of the tool. A new generation of abrasive grinding tools can be obtained by adding TEOS.

The addition of TEOS plays an important role, allowing the composite to dimensionally stabilize during processing and during tool use, which is a feature that will allow for greater performance and accuracy of the abrasive tool. At the same time, while vitrified bond was rearranged between the grains, the porosity of the composite increased, also improving its wear properties and reducing the surface roughness of the composite, allowing a better surface finish of the workpieces.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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