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The effect of graphite addition on the mechanical and tribological properties of pitch-based granular carbon composites

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Abstract The present work deals with the effect of graphite addition on selected mechanical and tribological properties of pitch-based granular carbon composites. Three pitches (a commercial impregnating coal tar pitch, an air-blown pitch and a thermally treated coal tar pitch) and anthracite particles as reinforcing material were used to prepare carbon composites to be tested as carbon brake pads. These carbon composites show good compression strength (from 25.8 to 94.2 MPa) but unstable and high friction coefficients (>0.5). Experimental results have showed that small amounts of graphite addition (2.5 and 5.0 wt%) lead to carbon materials with more stable and lower friction coefficient (<0.3). So, graphite addition promoted the reduction in the wear rate. Finally, compressive strength of carbon composites prepared with modified pitches significantly increases after addition of small amounts of graphite with values from 94 to 128.8 MPa.

Introduction

Carbon composites play an important role in brake systems due to their unique properties such as low coefficient of thermal expansion, high heat capacity, good thermal shock resistance, low weight and exceptional wear resistance [1]. Carbon composites are normally manufactured using a carbon fibre preform which is densified with a matrix precursor [2]. There are two main processing techniques of densification: The chemical vapour deposition of a hydrocarbon gas, CVD, and the successively liquid impregnation process with an organic precursor, resin or coal tar pitches [2, 3]. In both cases, precursors and processes were expensive leading to high-cost carbon materials. This fact limits their use to high-technological applications such as aeronautics, aerospace or military industry where no other materials are suitable. In conventional applications where high performance materials are not required the use of cheaper precursors and more simple fabrication processes could reduce the cost of carbon composites. In this way, granular carbons could be used as an alternative to carbon fibres for reinforcing materials [4, 5].

Coal tar pitches have been extensively used as carbon matrix precursors due to their low price, relatively high carbon yield and their ability to generate graphitisable carbons after carbonisation treatment [6]. Moreover, the carbon yield of the pitches can be improved and the porosity of the carbon material reduced by air-blowing [7] or thermal treatment [8]. It is generally accepted that pitch-air blowing yields cross-linked structures with bridge-bonded macromolecules through the formation of oxy-radicals [7], whereas pitch thermal treatment generated more planar and condensed molecules, usually with carbonaceous mesophase [8, 9]. Coal tar pitches consist of hundreds of chemical compounds with molecular mass ranging from a few hundred to several thousand atomic units [10]. The transformation of pitches into carbon materials (coke) requires a carbonisation process. The behaviour of the pitch during such heat treatment and consequently, the properties of the final carbon composite depend on the raw materials properties [11], the

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experimental conditions used and the properties of reinforcing carbon material mixed with the pitch [12–14].

Previous research carried out by our group has shown that carbon composites prepared with pitches and different granular carbons are an interesting alternative for conventional brake systems due to their compressive strength and wide range of friction coefficients with values from 0.12 to 0.76 depending on experimental conditions such as load applied and sliding speed [5]. However, carbon composites reinforced with anthracite show very unstable friction coefficient that could make difficult their industrial application [5]. Lubricant materials are used to reduce the friction and the wear in the sliding contact. The presence of the lubricating film prevents the direct contact between the two surfaces resulting in stable friction coefficients and lower wear rates. Traditionally, lubricants were externally added. However, self-lubricating materials are more desired as they were released automatically during their use [15]. In this sense, graphite was extensively used leading to better friction stability than other solid lubricants [16–19].

The main objective of the present work is to study the effect of graphite addition on the mechanical and tribological properties of pitch-based composites reinforced with anthracite. Small amounts of graphite (2.5 and 5.0 wt%) were added to the pitch/anthracite mixture. Carbon composites were characterised according to their open porosity, carbon yield, light texture, compressive strength, friction coefficient and wear rate. Experimental results were compared with those obtained from carbon composites without graphite addition.

Experimental

Raw materials and characterisation

Three pitches were used as carbon matrix precursors: a commercial impregnating coal tar pitch (D0), supplied by Industrial Química del Nalón S.A. (Asturias, Spain); a new pitch (G18) prepared by air-blowing of the same commercial pitch, D0, at 275 °C for 18 h and the third pitch (D3) was obtained by thermal treatment of D0 at 430 °C for 3 h. The detailed experimental conditions for the preparation of G18 and D3 are given elsewhere [20]. Prior to their use as carbon matrix precursors, the pitches were ground and sieved to a particle size <0.4 mm. Main characteristics of three pitches are summarised in Table 1.

Anthracite (AT) from the North of Spain was used as reinforcing carbon whereas graphite particles (GR) supplied by ISMAF (Bilbao, Spain) was used as self-lubricating material. Both carbons were ground and sieved to a particle size <0.1 mm. Table 2 shows the main properties of anthracite and graphite used in this work.

Table 1 Main properties of pitches: D0, D3 and G18

Pitch	Elemental Analysis (dry basis, wt%)					C/H[a]	SP[b]	CY[c]	TI[d]	MC[e]
	C	H	N	S	O					
D0	92.47	4.53	1.12	0.56	1.61	1.70	97	46.0	21.8	0
D3	94.05	3.98	1.11	0.46	1.05	1.98	169	69.4	51.6	30
G18	92.81	4.15	1.15	0.56	1.79	1.86	180	59.6	46.7	0

[a] Carbon/hydrogen atomic ratio

[b] Softening point (°C)

[c] Carbon yield (wt%)

[d] Toluene insoluble content (wt%)

[e] Mesophase content, determined by light microscopy (vol%)

Composite preparation

Carbon composites prepared in this work were classified into two types: Binary and ternary composites. Binary composites were prepared from mixtures of each pitch and anthracite whereas ternary composites were obtained from mixtures of pitch and two granular carbons (anthracite and graphite). The preparation of both kinds of materials involved three steps: the mixing of pitch/granular carbon systems, the moulding of the blend to obtain the green coke composites and the carbonisation up to 1,000 °C in order to transform the pitch into a carbon matrix (coque). The initial experimental conditions (moulding temperature, carbonisation heating rate or final temperature) were selected on the basis of the results obtained in previous works [4, 5]. A flow diagram of the experimental process is given in the Fig. 1.

Mixing process

The first step in the preparation of the binary or ternary carbon composite was the mixture of two or three components, respectively. In order to ensure adequate pitch fluidity and a good impregnation of the granular carbons, the mixtures were performed at temperatures of about 100 °C above the softening point of the pitches (Table 1). Thus, a temperature of 190 °C was used for D0 and 270 °C for G18 and D3 pitches.

For binary composites, the mixing process of two components was carried out at an initial pitch/granular carbon ratio of 25/75 in weight for D0/AT mixtures and 30/70 for D3/AT and G18/AT mixtures. These values were optimised in a previous work according with to criteria: (i) that the conformed pellet not deform when carbonised; (ii) that the non-deformed pellet presents the maximum compressive strength of each pitch/granular carbon mixture [5].

Ternary composites were initially prepared using the same pitch ratio as for binary composites (25 wt% for D0

Table 2 Main properties of anthracite (AT) and graphite (GR)

Granular carbon	Elemental Analysis (dry basis, wt %)					Moisture (wt%)	Ash (wt%)	CY[a]	d^{b}_{He}	d^{c}_{002}	Size particle distribution (vol%)		
	C	H	N	S	O						d_{75}	d_{50}	d_{25}
AT	82.04	1.69	0.95	1.00	2.96	3.41	11.37	90.6	1.70	3.52	75.7	35.7	14.2
GR	87.45	0.05	0.00	0.25	0.75	0.08	11.50	97.3	2.35	3.39	97.2	64.1	35.7

[a] Carbon yield (wt%)

[b] Helium density (g cm^{-3})

[c] Interlaminar space, determined by X-ray diffraction (Å)

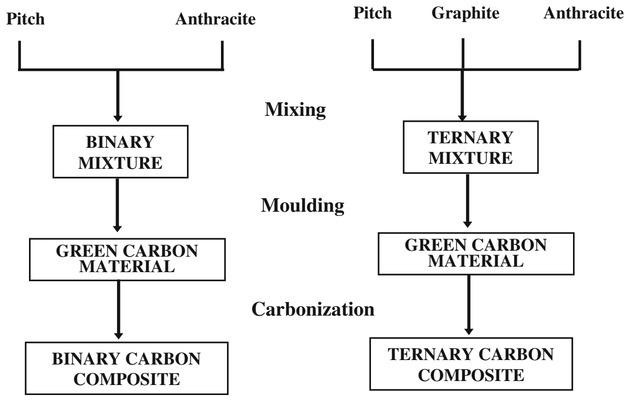

Fig. 1 Flow diagram of binary and ternary carbon composites preparation

and 30 wt% for D3 and G18 pitches). Graphite particles were added in 2.5 or 5.0 wt%, reducing the anthracite content in the same ratio. If the pellets deformed during carbonisation, new ternary composites were prepared reducing the pitch ratio by 5 wt%.

In each case, 100 g of mixture was prepared in a 0.5 L stainless steel reactor and heated in an electrical furnace under continuous stirring and a nitrogen atmosphere. Once the mixing process was accomplished, the mixture was sieved to below 1 mm.

Moulding process

The mixture was moulded in one uniaxial press. Ten grams of sample was placed in a stainless steel mould with an inner diameter of 30 mm and was heated to a temperature slightly below the softening point of the pitch in order to prevent the pellet from springing back on demoulding. For this reason, a small furnace was placed around the mould. The furnace was connected to a temperature controller and a thermocouple inserted inside the mould. According to previous works the temperature used was 40 °C for D0 and 140 °C for D3 and G18 mixtures [4, 5]. Once the desired temperature was reached, 125 MPa of uniaxial pressure was applied for 3 min. In all cases, the same pressure and moulding time were used and only the temperature was altered [4, 5].

Carbonisation process

Green coke composites were placed in a graphite crucible and covered with an inert material at elevated temperatures (zircon of particle size <0.1 mm). The crucible was placed in a horizontal furnace and the temperature was raised to 1,000 °C at a rate of 1 °C min^{-1}, under a nitrogen flow rate of 25 L h^{-1}. The final temperature was maintained for 30 min.

Characterisation of mixtures

Binary and ternary pitch/granular carbon mixtures were characterised by optical microscopy. Samples were embedded in an epoxy resin, polished with diamond solution (1 μm) and examined using an optical microscope fitted with a polarizer and a one-wave retarder plate to generate interference colours. Representative photographs of the polished samples were taken using oil-immersion objectives of 20, 50 and 100× magnification.

Characterisation of carbon composites

Carbon yield

The carbon yield of the carbon composites was determined from the weight variation after the carbonisation of the green coke composites at 1,000 °C in a tubular furnace using a heat rate of 1 °C min^{-1}, under nitrogen flow rate of 25 L h^{-1}. The final temperature was maintained for 30 min.

Theoretical and experimental carbon yields were compared in order to obtain an approximate idea of the pitch/granular carbon interactions. The theoretical values were obtained as a weight average of the carbon yields of the separated components, pitches, anthracite and graphite (Table 1).

Bulk density and volume variation

The bulk density of the green and carbonised materials was calculated from the weight and volume of the green and carbonised pellets, respectively. Shrinkage and expansion were determined from the variation in the volume of the green and carbonised pellets, using a calliper.

Apparent water density and open porosity

The apparent water density and open porosity of the green and carbonised composites were determined by water immersion in accordance with ASTM C20-83.

Real density

The real density of the green and carbonised materials was determined by helium pycnometry using a Micromeritics AccPyc 1330 apparatus.

Microstructural characterisation

The microstructure (optical texture, porosity and interfaces) of the composites was studied by optical microscopy. Pellets were cut in two pieces and embedded in an epoxy resin. The mounted samples were successively ground on two grades of silicon-carbide paper (600 and 1,200 grits) and polishes successively with diamond solution (1 μm). Microscopic analysis was conducted in a similar way to that described in Sect. 2.3.

Compressive strength

The compressive strength of carbon composites were evaluated in accordance with the ASTM C 695 standards using an Instron 5583 equipment. The results are quoted as the mean values obtained from three samples of each carbon composite.

Tribological properties of carbon composites

The tribological behaviour of carbon composites was evaluated from their friction coefficient and wear rate using a home built friction testing machine (Fig. 2). This equipment consists of a metallic disc of 22 cm external diameter and 15 cm internal diameter, connected to a motor. The sample (2–2.5 cm of diameter) was placed in a metallic holder coupled to a pneumatic system that pushes the pellet against the rotating disk. The speed of the disc, the normal load applied to the sample, the torque and the temperature of the samples were registered continuously. The friction coefficients were calculated using the following expression (1),

$$\mu = \frac{M \cdot 3 \cdot \left(R_{\text{ext}}^2 - R_{\text{int}}^2\right)}{2 \cdot F \cdot \left(R_{\text{ext}}^3 - R_{\text{int}}^3\right)} \tag{1}$$

where μ is the friction coefficient, M the torque, F the load applied to the pellet and R_{ext} and R_{int} the external and internal radio of the friction area, respectively. The wear rates were determined by measuring the weight variations of the carbon composite at different stages and at the end of each test. The wear was showed as weight loss by sliding distance and load applied ($\text{g}^{-1}\ \text{N}^{-1}$).

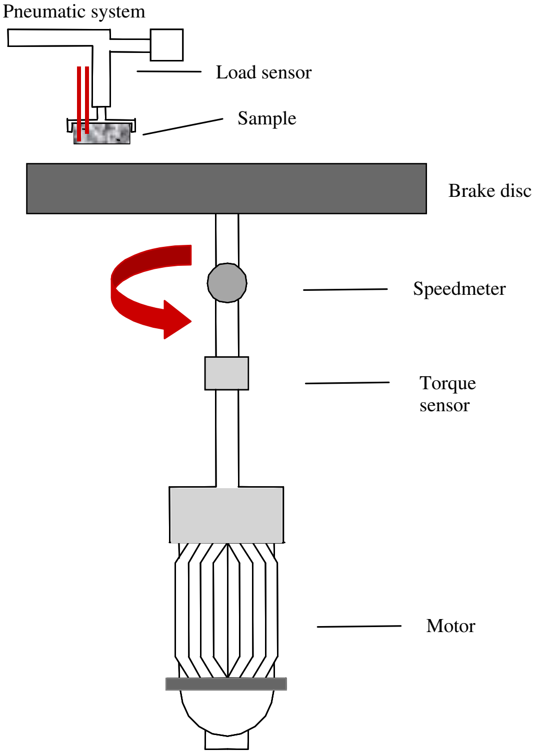

Fig. 2 Friction test equipment

Results and discussion

Characterisation of pitch/granular carbon mixtures

Binary mixtures

Careful study of the binary pitch/anthracite mixtures was performed elsewhere [13, 20]. Isotropic pitches (D0 and G18) lead to homogeneous mixtures (Fig. 3a and b). In case of thermally treated pitch (D3), some of the mesophase spheres (Fig. 3c, position MS) deformed or coalesced during their mixture with anthracite particles leading to more heterogeneous samples. Previous works have showed that mesophase deformation and coalescence during the mixture at the relatively low temperature used (270 °C) could be related to remarkably powerful interactions between the primary quinoline insoluble (QI) that initially surrounded mesophase spheres and the anthracite particles [20].

Ternary mixtures

Figure 4a shows a representative micrograph of graphite used in this work. It could be observed that graphite particles are laminar with a light texture of flow domains as a result of their highly ordered microstructure. Representative micrographs of ternary mixtures are showed in Fig. 4b

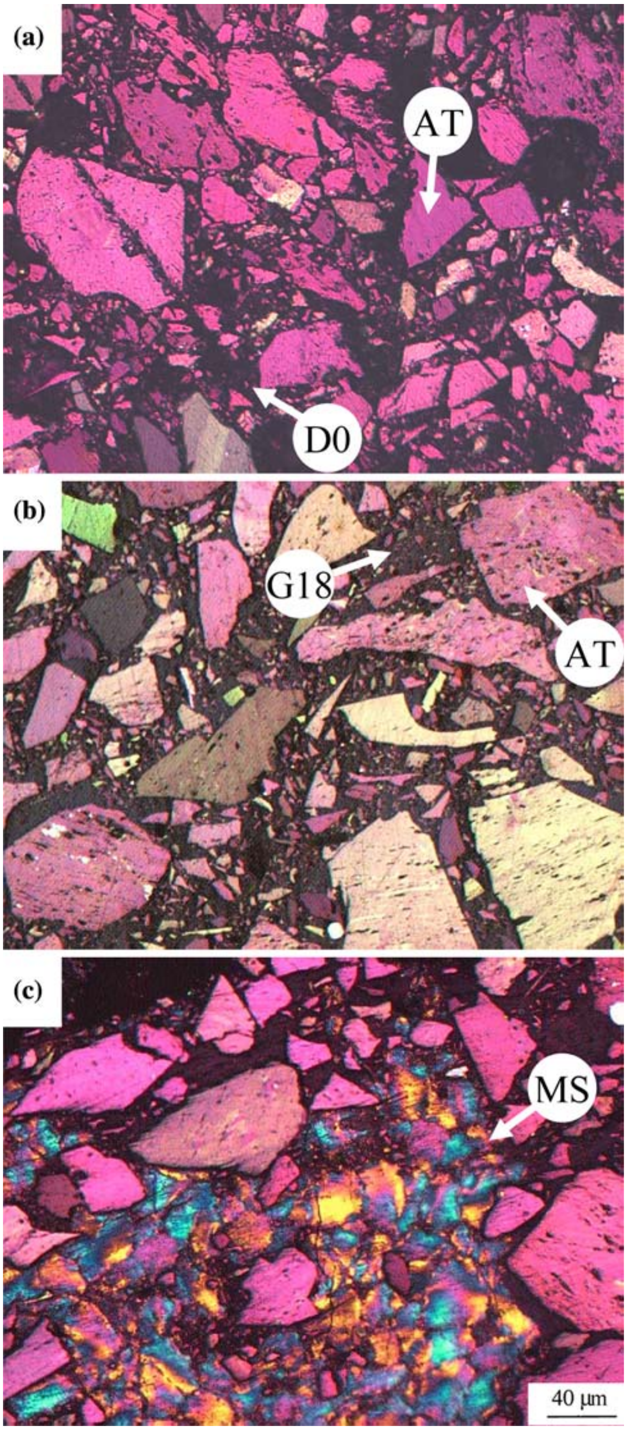

Fig. 3 Optical micrographs of binary mixtures: (**a**) G18/AT, (**b**) D0/AT and (**c**) D3/AT

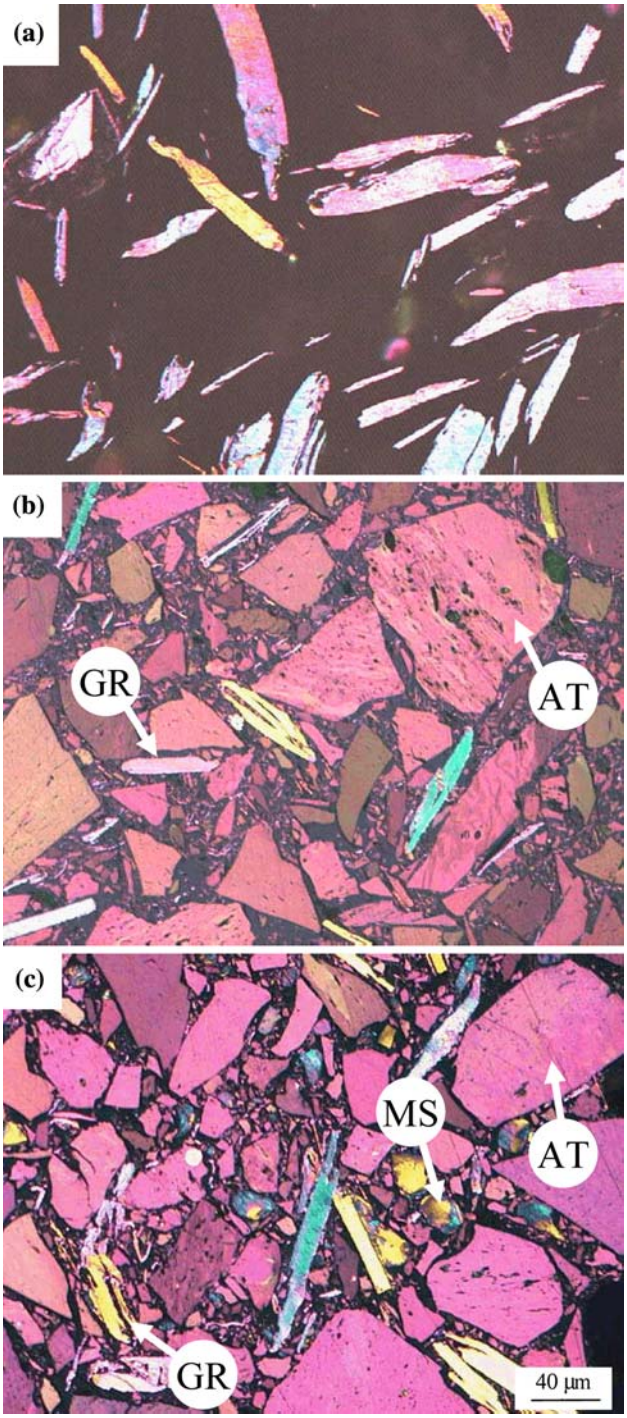

Fig. 4 Optical micrograph of (**a**) graphite and ternary mixtures (**b**) G18/AT/GR and (**c**) D3/AT/GR

(with G18 pitch) and 4c (with D3 pitch). Careful observation shows that graphite was homogeneously distributed between the pitch and the anthracite particles. Moreover, comparison with Fig. 4a shows that some graphite particles reduce their size during their mixture. Size reduction could be due to the hardness of anthracite particles that strike with graphite during the mixing process. Figure 4c shows that mesophase particles (Fig. 4c, position MS) of thermally treated pitch, D3, appear homogenously distributed around the samples. The mesosphere deformation and coalescence produced during mixing process of D3 and anthracite (Fig. 3c, position MS) was significantly reduced after graphite addition (Fig. 4c) leading to more homogeneous mixtures.

Carbon composites preparation

Binary and ternary carbon composites were prepared as was showed in Fig. 1. During carbonisation, pitches exhibited important transformations as a result of which it was necessary to optimise the pitch content to ensure that shape of pellet is maintained. According to previous works [5], the initial pitch ratio used for binary and ternary composites was 25 wt% for D0 mixtures and 30 wt% for mixtures with D3 and G18 pitches. However, ternary composites prepared with D3 and G18 pitches deform during carbonisation and the amount of pitch was reduced by 5 wt%. The same pitch ratio was used for binary composites even though this was not the optimum pitch/granular carbon ratio to facilitate the comparison of the binary and ternary composites. So, finally 25 wt% of pitch was used for the preparation all the composites.

Previous works have shown that as the presence of carbons seriously alters the pyrolysis of pitches [12, 13]. Graphite produces an important delay in pyrolysis which might explain the tendency of the composites to swell, as the pitch remains softer for a longer period of time and at higher temperatures.

Characterization of carbon composites

Carbon composites were characterised in order to study the effect of graphite addition on their structural, mechanical and tribological properties.

Carbon yield

Addition of graphite particles slightly increases the carbon yield of the final material according to the high carbon yield of graphite (Table 2). Moreover, it was interesting to compare the difference between the experimental and theoretical carbon yields ($r_e - r_t$) of binary and ternary carbon composites (Table 3). According to previous works, all values show in Table 3 are positive due to the fact that the experimentally detected carbon yields of the pitches are higher in the presence of granular carbons [5, 13]. In general, similar $r_e - r_t$ values were obtained (from 4.19 to 5.83 wt%). In all cases, it could be established that graphite addition slightly decreases the difference between experimental and theoretical carbon yields.

Table 3 Experimental and theoretical carbon yields differences ($r_e - r_t$) in the binary and ternary carbon composites (wt%)

Carbon composite	Pitch		
	D0	D3	G18
Binary	5.83	5.50	5.04
Ternary (2.5 wt% of GR)	4.77	4.90	4.49
Ternary (5 wt% of GR)	4.76	4.19	4.36

Volume variation

Graphite particles were not affected by thermal treatment. However, it is well known that pitches tend to swell when they undergo thermal treatment between 300 and 450 °C and then shrink when treated at about 700 °C. On the other hand, anthracite particles considerably shrink at 600–700 °C. So, considerable variations in the volume of carbon composites would be produced after the carbonisation of the pellets. Table 4 summarises the volumetric changes of carbon composites. All pellets shrink during carbonisation, with volume variations from 14.3 vol% for D0/AT with 2.5 wt% of GR to 17.9 % for D3/AT with 2.5 wt% of GR. This fact could be due to the shrinkage of the main component, the anthracite particles. It could be observed in Table 4 that graphite addition does not produce important variations in the volume shrinkage (binary and ternary composites show similar values, from 14.3 to 17.9 vol%).

Open porosity

Volume and weight changes produced during carbon composites carbonisation will originate porosity of pellets. Table 5 shows the open porosity of binary and ternary carbon composites. Generally, the porosity developed during carbonisation is elevated (15.9–37.0 vol%), specially when commercial coal tar pitch is used (from 27.6 to 37.0 vol%). The use of treated pitches significantly reduces

Table 4 Volume variations on carbonisation of binary and ternary carbon composites (vol%)

Carbon composite	Pitch		
	D0	D3	G18
Binary	−16.9	−15.7	−16.5
Ternary (2.5 wt% of GR)	−14.3	−17.9	−16.4
Ternary (5 wt% of GR)	−15.3	−16.8	−17.1

Table 5 Open porosity of binary and ternary composites (vol%)

Carbon composite	Pitch		
	D0	D3	G18
Binary	37.0	25.9	26.2
Ternary (2.5 wt% of GR)	27.6	15.9	19.3
Ternary (5 wt% of GR)	29.0	18.7	19.9

the porosity of corresponding binary or ternary carbon composites due to the high carbon yield of D3 and G18 pitches (Table 1). Also, graphite addition produces a remarkable reduction of pellets porosity, especially when thermally treated pitch was used as carbon matrix precursor (from 25.9 to 15.9 vol% after addition of 5 wt% of graphite). Lower porosity of ternary composites could be related to the production of more homogeneous mixtures after graphite addition. It was shown that mesosphere deformation and coalescence produced during mixing process of D3 and anthracite was significantly reduced after graphite addition leading to more homogeneous mixtures.

Microstructure analysis

After the carbonisation of pellets, pitches were transformed in the corresponding carbon materials. Previous works have showed that optical texture of carbon matrix significantly depends on the pitch properties and interactions with the granular carbon used [12, 13]. Figure 5 shows optical micrographs of binary and ternary composites. Careful analysis of samples shows that there are no important differences between the optical texture of binary and corresponding ternary carbon composites. In the binary composites with thermally treated pitch (Fig. 5a), the light texture from the mesophase (position A) and the isotropic phase (position B) was different, leading to a more heterogeneous sample. However, in case of corresponding ternary composites with a 5 wt% of graphite (Fig. 5b) a more homogenous sample was obtained. This fact is related to better pitch distribution after graphite addition (Fig. 4). In case of anthracite and isotropic pitches (D0 and G18), homogenous pellets with a low size texture throughout the pellet were obtained. Figure 5c and d shows representative micrographs of binary and ternary composites, respectively, prepared with G18 pitch.

Mechanical properties

Mechanical properties of carbon composites were evaluated from their compression strength. Figure 6 shows the compressive strength of binary and corresponding ternary composites. It could be noted that the compressive strength of binary composites decreases with the use of modified pitches (D3 and G18). This occurs in spite of the high carbon yield of treated pitches (Table 1) and the lower porosity of pellets (Table 5). Previous works carried out by our research group showed that treated pitches produce carbon materials with a lower porosity and a higher carbon yield [5]. However, the compressive strength of the corresponding materials is not always higher. There are other factors that may affect the mechanical properties of carbon composites, such as the quality of the mixture of the two components or the pitch/granular carbon interfaces. However, it was interesting to note that graphite addition significantly increases the mechanical resistance of carbon composites (Fig. 6), especially when treated pitches were

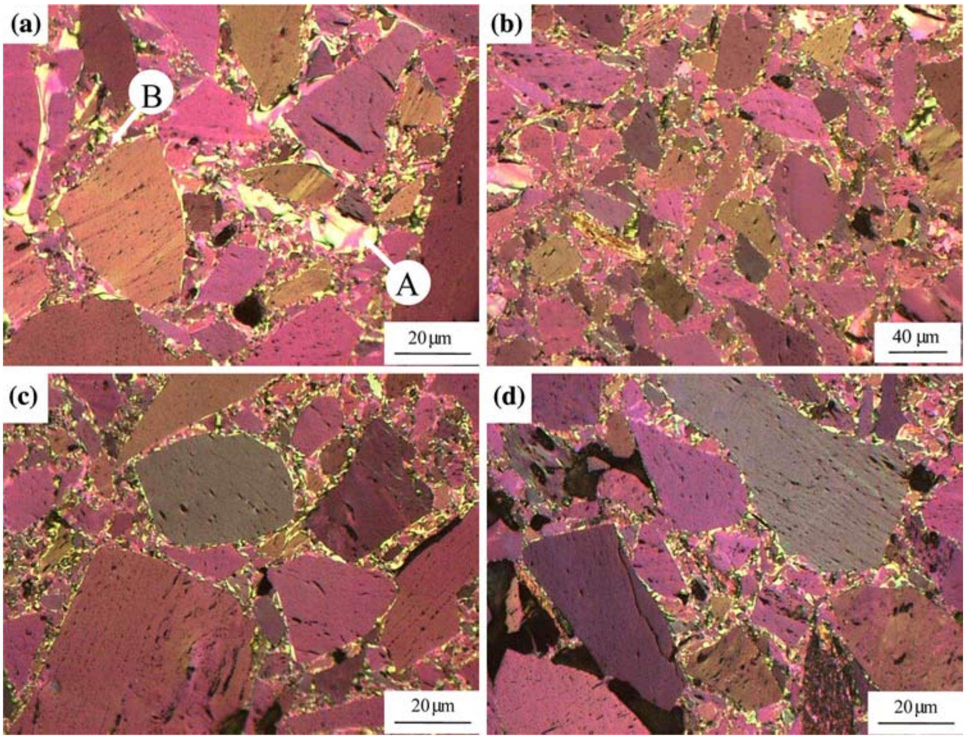

Fig. 5 Optical micrographs of binary and corresponding ternary composites: (**a**) D3/AT; (**b**) D3/AT/GR; (**c**) G18/AT and (**d**) G18/AT/GR

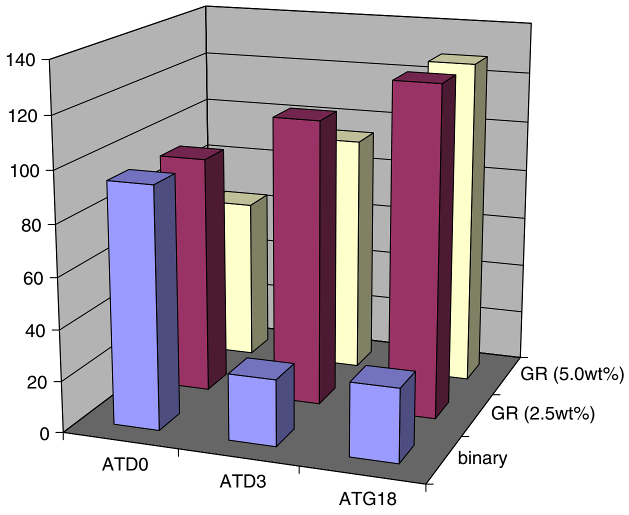

Fig. 6 Compressive strength of binary and ternary carbon composites

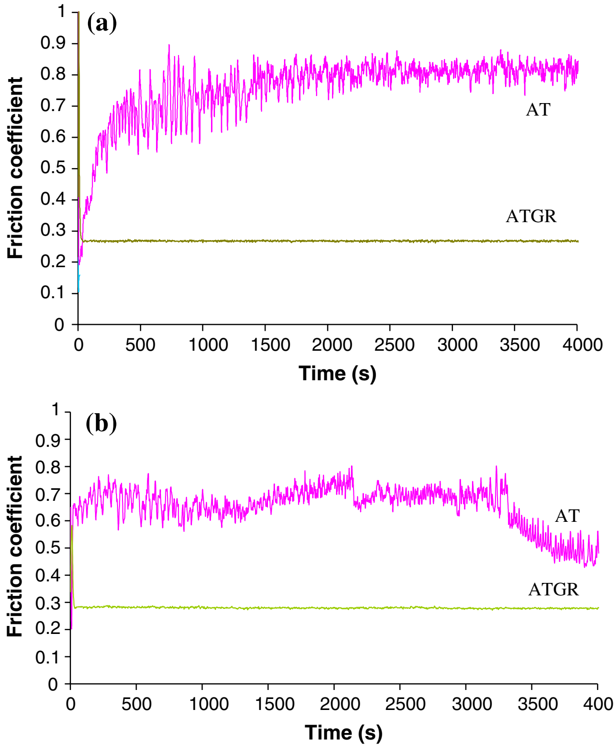

Fig. 8 Friction coefficients of binary and ternary carbon composites with: (**a**) D3 and (**b**) G18 pitches

used as carbon matrix precursors. This enhancement was not related with the decreases in the porosity promoted by graphite addition (Table 5) because, in case of D0 pitch, porosity significantly decreases after addition of graphite but not important changes were produced on the compressive strength (Fig. 6). On the other hand, no important differences were observed in the microstructure of binary and ternary composites (Fig. 5). So, other factors should be responsible of this effect. Previous works carried out by Yasuda et al. [21] have showed that graphite addition significantly improves the mechanical properties of carbon composites leading to a plastic behaviour In order to understand this effect, samples obtained after compression test of pellets were analysed by light microscopy (Fig. 7). Analysis of samples shows that cracks originated during compression strength flow through the anthracite particles and the carbon matrix. However, it is interesting to note that these cracks stop their propagation on the graphite surfaces perpendicularly oriented to their propagation (Fig. 7, position A). It is known as the elevated anisotropy of graphite: soft in the parallel direction to their graphitic planes but very strong on the perpendicular direction. Thus, the homogenously distribution of graphite in the samples leads to carbon composites with graphite particles oriented in all directions that delays crack propagation increasing the mechanical resistance of ternary carbon composites.

Tribological properties

The tribological behaviour of carbon composites was evaluated from the friction coefficient and wear rate. Tests were performed by sliding the carbon composites against a commercial metallic car brake. Friction tests were carried out using a variety of conditions, and a wide range of loads (98, 147 and 196 N) and sliding speed (400, 600 and 800 rpm) were examined. Figure 8 shows the friction coefficient evolution of binary and ternary composites with anthracite and D3 (Fig. 8a) or G18 pitch (Fig. 8b). Binary

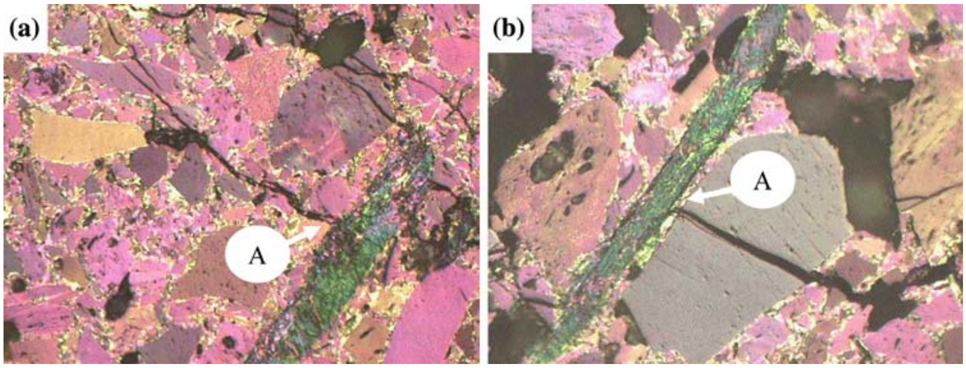

Fig. 7 Optical micrographs of carbon composites fragments obtained after compression tests

Table 6 Wear rate of binary and ternary composites (g^{-1} N^{-1})

Carbon composite	Rate (rpm)	Load (*N*)	Wear rate (g^{-1} N^{-1} x 10^{-9})
Binary with D0 pitch	600	147	2.59
Ternary (5 wt% of GR) with D0 pitch	600	147	2.17
Binary with D0 pitch	600	98	1.72
Ternary (5 wt% of GR) with D0 pitch	600	98	2.32
Binary with D3 pitch	600	98	5.45
Ternary (5 wt% of GR) with D3 pitch	600	98	5.75
Binary with D3 pitch	400	98	3.45
Ternary (5 wt% of GR) with D3 pitch	400	98	1.34
Binary with G18 pitch	600	98	13.4
Ternary (5 wt% of GR) with G18 pitch	600	98	5.83
Binary with G18 pitch	500	98	6.70
Ternary (5 wt% of GR) with G18 pitch	500	98	3.33

composites show high irregularity in their friction coefficient. It could be observed that graphite addition reduces the friction coefficient of carbon composites and makes it more stable due to the ability of graphite to act as a self-lubricant. These results were according with that obtained previously by other authors [16–19].

Wear of carbon composites during the sliding is a very important factor because it determines the life of the material. Table 6 shows the wear rates of binary and ternary composites obtained under different experimental conditions. Wear was presented as weight loss by sliding distance and load applied (g^{-1} N^{-1}). Wear of binary composites increases with the rate of rotating disc and the load applied. Generally, wear rates of ternary composites were lower than in the corresponding binary composites, especially when treated pitches were used as carbon matrix precursors.

Conclusions

The mesosphere deformation and coalescence produced during mixing process of D3 and anthracite was significantly reduced after graphite addition leading to more homogeneous mixtures.

Binary carbon composites show compression strength values from 25.8 to 94.2 MPa. Graphite addition significantly increases the compressive strength of ternary composites prepared with treated pitches leading to values from 94 to 128.8 MPa.

Cracks originated during compression strength of ternary carbon composites stop their propagation on the graphite surfaces perpendicularly oriented to their propagation increasing the compressive strength of ternary composites.

Binary composites reinforced with anthracite show unstable and high friction coefficients (>0.5). Addition of graphite particles lead to carbon composite with more stable and lower friction coefficient (<0.3). So, graphite addition promoted the reduction in the wear rate.

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