

Study of the Influence of *Nejayote* and Other Additives on the Cohesive Strength and Electric Properties of Carbon Black Agglomerates

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ABSTRACT: An investigation was carried out on the influence of *nejayote* and other additives (water, potassium lignosulfonate, and furfuryl alcohol), on the cohesive strength and electrical properties of carbon black agglomerates. The thermal stability of carbon black agglomerates with potassium lignosulfonate and *nejayote* was similar. The influence of *nejayote* and potassium lignosulfonate on the cohesive strength of carbon black agglomerates was also similar but the electrical properties were different. The carbon black agglomerates with *nejayote* have a lower electrical conductivity. The influence of water

on the cohesive strength of carbon black agglomerates was not significant but it was important in their electrical conductivity. *Nejayote*, a waste by-product from the corn-processing industry, appears as a potential agglutinant in the carbon black pelletization process. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 90: 3965–3972, 2003

Key words: additives; cohesion; carbon black agglomerates; electrical conductivity; agglutination

INTRODUCTION

Carbon black (CB) is a complex material. It is widely used in the plastic, paint, and rubber industries.¹ Non-rubber applications of CB are generally based on its absorption of visible and ultraviolet light; its electrical conductivity; and to a minor extent, on its high surface area and adsorptive capacity. Many aspects of the behavior of CB are based on its electrical properties. Electrical conductivity is important in many rubber and plastic materials, including antistatic applications, wire and cable sheathing, shielding against electromagnetic interference, and positive coefficient materials.² It can be added to some insulators to give them a controlled electrical conductivity.

The application of CB in pellet form in different technological sectors depends on, among other factors, the grain size distribution and hardness of the pellets. All these parameters are associated with the agglutination process, which depends on the quantity of water and additive used in the pelletization process. Potassium lignosulfonate (LSP) is one of the most important additives, which is obtained from spent wood-pulp liquors

after chemical treatment.³ Other natural sources can also be used as agglutinants in the CB pelletization process. For instance, certain technologies in the corn-processing industry generate waste by-products, which could be combined with LSP in the agglutination of CB. However, this possibility needs to be evaluated.

Alkaline cooking of corn in a saturated solution of lime [Ca(OH)₂] followed by its steeping and washing is a well-known procedure for production of corn-based food in Latin American countries such as Mexico and Guatemala.⁴ This process is known as *nixtamalization*. The cooking liquor and the washing water, the so-called *nejayote*, are important sources of pollution.^{5,6} *Nejayote* is characterized by a high organic matter content and it is especially rich in polysaccharides, that is, about 75% of nonstarch polysaccharides from alkaline-soluble hemicelluloses, known as “corn hull gums,”^{7–10} which have potential functional properties, such as thickening, emulsifying, stabilizing, and extending.⁷

The highly alkaline cooking/steeping liquor (pH 11–12, *nejayote*) is rich in corn solids and excess of lime. It is a waste by-product of both traditional *nixtamalization* and industrial instant flour production processes. Manufacturing plants dedicated to the corn *nixtamalization* process usually integrate large waste and wastewater treatment facilities to meet environmental regulatory requirements. *Nejayote* has a chemical oxygen demand (COD) of about 25,000 mg/L, a biological oxygen demand (BOD) of 8100 mg/L, and a suspended solids content of 20,000 mg/L.⁸ In addition to

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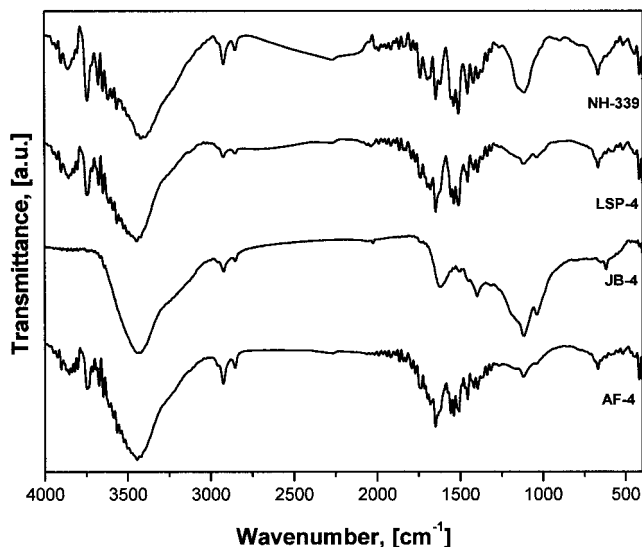


Figure 1 Infrared spectra of carbon black and its agglomerates with potassium lignosulfonate, *nejayote*, and furfuryl alcohol as additive.

a high COD and BOD, *nejayote* contains a concentration of nitrogen and phosphorous of nearly 310 and 180 mg/L, respectively.¹¹ As a result, practical uses of that effluent would be both environmentally and economically desirable. The present report represents an effort oriented in this sense, particularly using *nejayote* as a source of gums in the agglutination process of CB.

EXPERIMENTAL

Materials

To produce *nejayote* 500 g of maize kernel Bimbo, produced in Mexico, was boiled for 40 min in a 2 wt % calcium hydroxide solution and steeped from 95 to 20°C for a period of 14 h to produce *nixtamal*. The cooking liquor and the washing water from *nixtamal* were pooled and constituted the *nejayote*. This material was not neutralized.

The NHUMO Corp. (Mexico) provided LSP solution at 5%, fluffy CB N-339, and pelletized CB N-234. The CB morphological properties (mean particle diameter, surface area, and mean aggregate diameter) from CB N-339 and CB N-234 were previously reported.¹² Furfuryl alcohol (FA; Merck, Darmstadt, Germany) was purified, as previously reported.¹³

Methods

To prepare agglutinates of CB N-339, samples of fluffy CB N-339 were weighed (0.5 g), and controlled amounts of additives (water, LSP, *nejayote*, and FA) were added. The substances were mixed in a beaker and stirred using a magnetic stirrer at 60°C, to produce a paste of the agglomerate. At the end of the

mixing process, a spatula was used until no obvious large agglomerates were detected.

Samples of CB N-234 were depelletized to obtain a fluffy material and improve the homogeneity of the compacts. CB samples (0.1 g) in the form of compact disks (diameter 11 mm) were prepared by compressing aggregates of fluffy CB N-339 and N-234 depelletized in a testing machine at a pressure of 360 kg/cm² for 20 min. The sample weight and the applied pressure was kept constant because the manner in which the sample is packed can drastically affect its behavior during the cracking test. To study the influence of humidity on the electrical properties of the CB agglomerates, the samples were wetted in an autoclave and the percentage of adsorbed water was determined by gravimetry.

Measurements

Hardness test

A texture analyzer (Stable Microsystems, Ltd., UK) was used to measure the point at which the samples of compacted CB cracked. A TA.XT2 texturometer with a 25-kg capacity and 1 g sensitivity was used for the tests. All the samples were tested at room temperature. Cracked strengths were measured for compact disks covering a range of volume agglutinate/CB N-339 mass ratio [$r(Q/NHUMO)$]. The reported measurements are the average of 10 repetitions of each test. Statistical analysis procedures were applied to correlate the measured cracked strengths with the $r(Q/NHUMO)$ parameter.

Electrical resistivity measurement

The electrical resistivity was measured with a four-point probe technique. The method was based on the application of current and measurement of the resulting voltage. A geometrical correction factor for resistivity measurements on a circular specimen¹⁴ was made. A digimatic Mitutoyo micrometer ($X = \pm 0.001$ mm) was used to calculate the thickness (X , mm) of the sample. Before the measurements, all the samples were kept in a desiccator over silica gel, and the conductivity was measured in dry air at 22°C.

IR data collection

IR spectra of samples were recorded in KBr pressed disks on a Vector 22 FTIR spectrophotometer (Bruker Instruments, Billerica, MA).

Thermal analysis

Differential scanning calorimetric (DSC) data was obtained using a Perkin-Elmer Pyris-1 calorimeter (Perkin Elmer Cetus Instruments, Norwalk, CT) in the range 30–400°C at a heating rate of 5°C/min in a

TABLE I
Frequency (in cm^{-1}) of the Observed IR Absorption Bands
for Carbon Black and Its Agglomerates

NH-339		LSP-4		JB-4		AF-4	
3748	ν OH	3856	ν OH			3854	ν OH
3674	ν OH	3745	ν OH			3746	ν OH
3649	ν OH	3674	ν OH			3674	ν OH
3615	ν OH	3648	ν OH			3649	ν OH
3590	ν OH	3589	ν OH	3443	ν OH	3446	ν OH
3566	ν OH	3565	ν OH				
3420	ν OH	3446	ν OH				
2921	ν CH	2922	ν CH	2924	ν CH	2925	ν CH
2852	ν CH	2856	ν CH	2856	ν CH	2850	ν CH
1742	ν C=O	1741	ν C=O			1738	ν C=O
1699	ν C=O	1681	ν C=O	1699	ν C=O	1690	ν C=O
1650	$\delta\text{H}_2\text{O}$	1649	$\delta\text{H}_2\text{O}$	1620	$\delta\text{H}_2\text{O}$	1650	$\delta\text{H}_2\text{O}$
1558	ν C=C	1558	ν C=C			1559	ν C=C
1541	ν C=C	1540	ν C=C			1540	ν C=C
1511	ν C=C	1512	ν C=C			1509	ν C=C
1457	δCH_2	1457	δCH_2			1457	δCH_2
1421	δCH_2	1420	δCH_2			1419	δCH_2
1395	δCH	1397	δCH_2	1400	δCH_2	1397	δCH_2
		1208	$\nu^s \text{SO}_3^{2-}$				
1115	ν C—C	1111	ν C—C	1118	ν C—C	1120	ν C—C
		1033	$\nu^s \text{SO}_3^{2-}$				
669	γ OH	670	γ OH	621	γ OH	669	γ OH
420	γ C—OH	420	γ C—OH	421	γ C—OH	419	γ C—OH

nitrogen atmosphere. Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) data were obtained using a DuPont Instrument DTA 1600

(DuPont, Boston, MA) in an oxygen atmosphere at a heating rate of $5^\circ\text{C}/\text{min}$. Samples weighing between 5 and 10 mg were used.

TABLE II
Influence of Different Additives on the Cohesive Strength and Electric Properties of CB Agglomerates

Exp.	$r[\text{Q}/\text{m}(\text{NHUMO})]^a$ (mL/g)	Density (ρ) (g/cm^3)	Cracked strength (F) ^b (g/mm)	Electric conductivity (S) ($\Omega^{-1} \text{cm}^{-1}$)
NH-339	0.0 ^c	0.0613	40 ± 6	0.25
NH-234	— ^d	0.1055	50 ± 10	0.44
LSP-1	1.2 ^e	0.0654	70 ± 12	0.24
LSP-2	2.5 ^e	0.1003	130 ± 7	0.33
LSP-3	15.0 ^e	0.1101	500 ± 15	0.65
LSP-4	25.0 ^e	0.1152	600 ± 30	0.80
LSP-5	40.0 ^e	0.1234	$716,0 \pm 20$	0.99
LSP-6	50.0 ^e	0.1366	800 ± 60	0.65
JB-1	1.2 ^f	0.0723	61 ± 5	0.15
JB-2	2.5 ^f	0.0925	140 ± 10	0.27
JB-3	15.0 ^f	0.1179	400 ± 20	0.38
JB-4	25.0 ^f	0.1245	550 ± 20	0.52
JB-5	40.0 ^f	0.1912	649 ± 35	0.73
JB-6	50.0 ^f	0.2818	740 ± 25	0.24
H ₂ O-1	25 ^g	0.0969	50 ± 10	0.25
H ₂ O-2	50 ^g	0.0993	50 ± 5	0.28

^a $r[\text{Q}/\text{m}(\text{NHUMO})]$: volume additive/CB N-339 mass ratio.

^b The reported measurement is the average of 10 repetitions of each test.

^c Compacted spheres of fluffy CB N-339 without additive.

^d Pelletized CB N-234 sample provided by NHUMO Corporation.

^e Additive: potassium lignosulfonate at 5% wt.

^f Additive: *Nejayote*.

^g Additive: water.

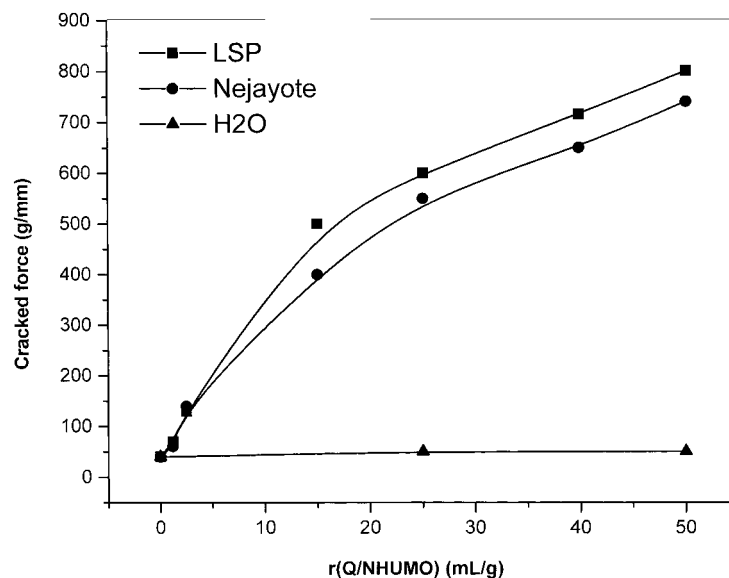


Figure 2 Cracked force versus $r(Q/NHUMO)$ data for the potassium lignosulfonate, *nejayote*, and water as additives.

X-ray dispersed-energy spectroscopy (EDS)

EDS spectra were recorded on pressed CB disks using a Noran Analytical system coupled to a SEM microscope (JEOL, Peabody, MA) operated at an accelerating voltage of 20 kV. Carbon and oxygen were not considered in the quantitative analyses of the detected elements.

RESULTS AND DISCUSSION

On the nature of cb agglomerates

The most important IR absorption bands detected for CB and its agglomerates (LSP-4, JB-4, AF-4) are presented in Figure 1 and Table I. Additional details on the studies samples are reported in Table II.

The assignment of bands was performed by comparing the IR spectra of the agglomerates with those corresponding to the organic additives. It is well known that CB has surface acidic functional groups, particularly carboxylic and phenolic ones.^{15–17}

The IR spectra of CB N-339 and its agglomerates are similar (see Fig. 1). However, the strong IR absorption band at 3420 cm^{-1} for CB shifts to $3440\text{--}3445\text{ cm}^{-1}$ in

the agglomerates, indicating the occurrence of certain interactions of the additives with CB. That interaction between the additive and the CB surface is believed to be attributable to hydrogen bonding and van der Waals forces. That type of interaction could be the result of intra- and intermolecular hydrogen bonding between the OH groups of phenolic ($3700\text{--}3590\text{ cm}^{-1}$) and carboxylic ($1740\text{--}1755\text{ cm}^{-1}$) structures that are present on the CB surface. The broad absorption band at 669 cm^{-1} was assigned to the out-of-plane deformation of the OH group as evidence of the occurrence of strong hydrogen bonding,¹⁸ whereas the absorption band at 410 cm^{-1} was attributed to the in-plane bending of the ring C—OH bond also attributed to hydrogen bonding.¹⁸ The JB-4 agglomerates do not present the IR absorption band assigned to carboxylic and phenolic groups (see Table I and Fig. 1) from the additive because the *nejayote* solution is basic (pH = 11.5). We did not observe the presence of the IR absorption band of the carboxylate ion, probably because the concentration of carboxylic groups on the surface of CB was not high. The FA-4 sample presents IR bands of FA with slight changes in their frequency

TABLE III
Influence of Furfuryl Alcohol on the Cohesive Strength and Electric Properties of Carbon Black Agglomerates^a

Exp.	Density (g/cm ³)	Percentage (in volume) of furfuryl alcohol	Cracked force (g/mm)	Electric conductivity (S) ($\Omega^{-1}\text{ cm}^{-1}$)
FA-1	0.1542	1	50 ± 5	0.25
FA-2	0.1560	4	70 ± 12	0.26
FA-3	0.1590	17	250 ± 25	0.35
FA-4	0.1594	29	660 ± 15	0.39

^a Mass of carbon black = 1 g. All experiments were carried out at $T = 60^\circ\text{C}$.

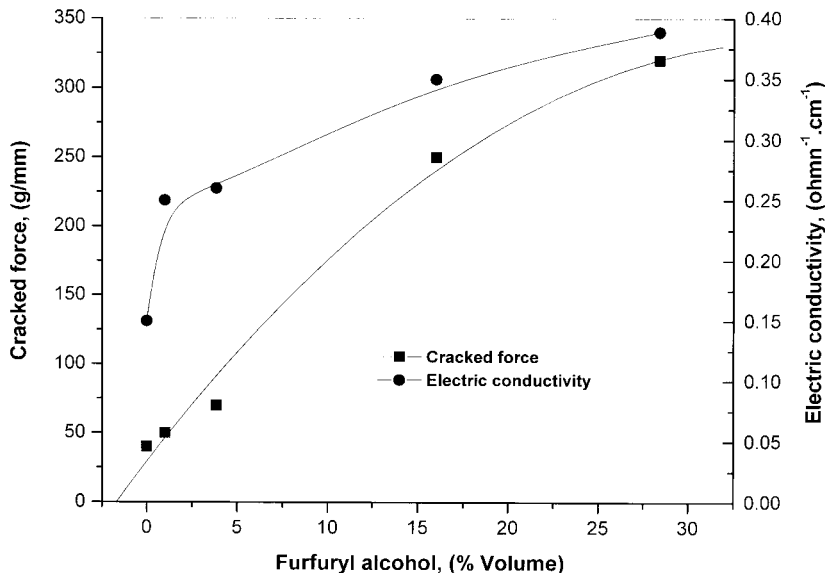


Figure 3 Cracked force and electrical conductivity behavior for furfuryl alcohol as additive.

and intensity, suggesting the occurrence of certain interaction between FA and CB.

Cohesive strength of CB agglomerates

The experimental conditions and the corresponding results concerning the influence of *nejayote*, LSP, and H₂O on the density, cohesive strength, and electrical properties of CB agglomerates are presented in Table II.

When any amount of additive was added to CB, it caused agglomerate formation, some of which seemed quite strong and difficult to eliminate. The addition of

greater amounts of additive (LSP and *nejayote*) to CB resulted in compacts with both high density and cracked strength. The critical cracked strengths for rupturing were higher for samples of compacted CB with higher density, whereas lower-density samples exhibited a high degree of dispersion in their mechanical resistance. This implies that the cohesive strength of samples increases with the compacted CB density. The cohesive strength of these compacts increased in the material when wet. The van der Waals attraction¹⁹ and other interaction forces are present in these CB agglomerates and could play an important role in the

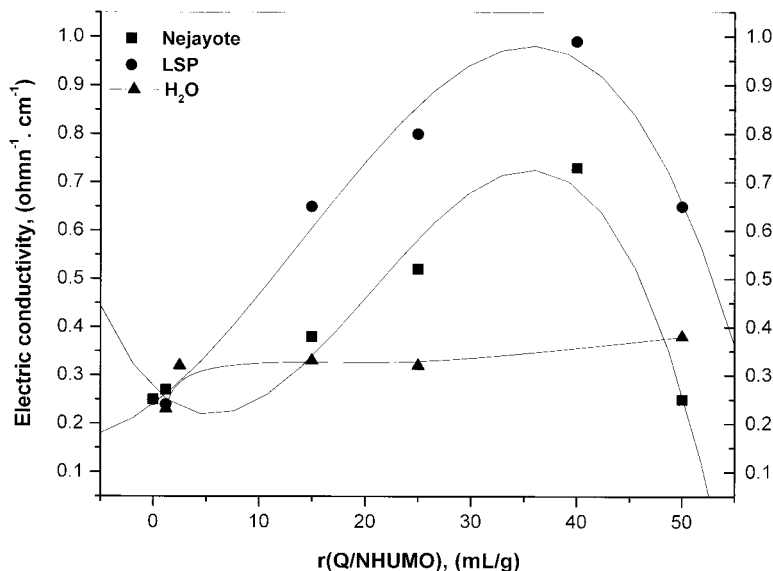


Figure 4 Electrical conductivity of carbon black agglomerates as a function of $r(Q/NHUMO)$ from potassium lignosulfonate, *nejayote*, and water as additives.

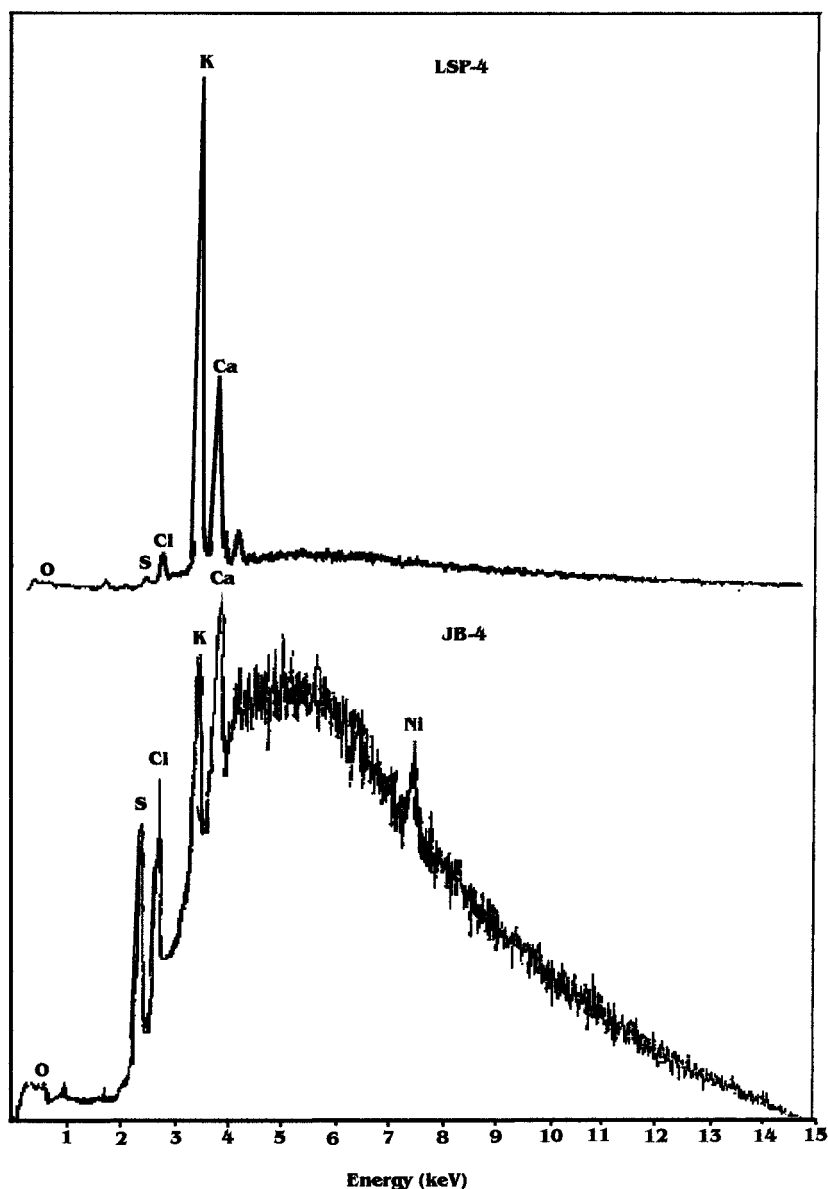


Figure 5 EDS spectra of LSP-4 and JB-4 samples.

cohesiveness of wet CB.^{20,21} However, water does not have an important role in promoting agglomeration. Water is an ineffective additive in production of high-density and compaction agglomerates.

The cracked force versus $r(Q/NHUMO)$ data, obtained for the LSP, *nejayote*, and water as additives tested are shown in Figure 2. It can be seen that the cracked force for the LSP and *nejayote* as additives are in the same general range, although it is different for water. Under the same experimental conditions (see Table II), water produces CB agglomerates of low density and rupture strength compared with those of LSP and *nejayote*.

The other additives used were different mixtures of FA and water (see Table III). On increasing the amount of FA added, the agglomerates had a higher density and the rupture force (see Fig. 3), which can be explained as a crosslinking effect of FA to form a resin.

FA in the presence of an aqueous solution of Brönsted acids and iodine reacts to give ramified resins that may even reticulate.^{13,22-26} It is well known that CB, having surface acidic functional groups such as carboxyl and phenolic groups, undergoes general chemical reactions. The carboxyl groups on the surface of CB have both a higher acidity and ability to initiate the polymerization of vinyl monomers^{15,16} and other species.¹⁷

The polymerization mechanism of FA in the presence of carboxylic groups on the surface of CB must be analogous to that reported by us for the polymerization of this monomer in the presence of TFA and iodine,^{13,24,25} or it is possible that thermal resinification of FA could take place. Investigations of polymerization of FA by the carboxyl group of CB and the grafting of polyfurfuryl alcohol onto CB surface are now in progress. The importance of this additive is

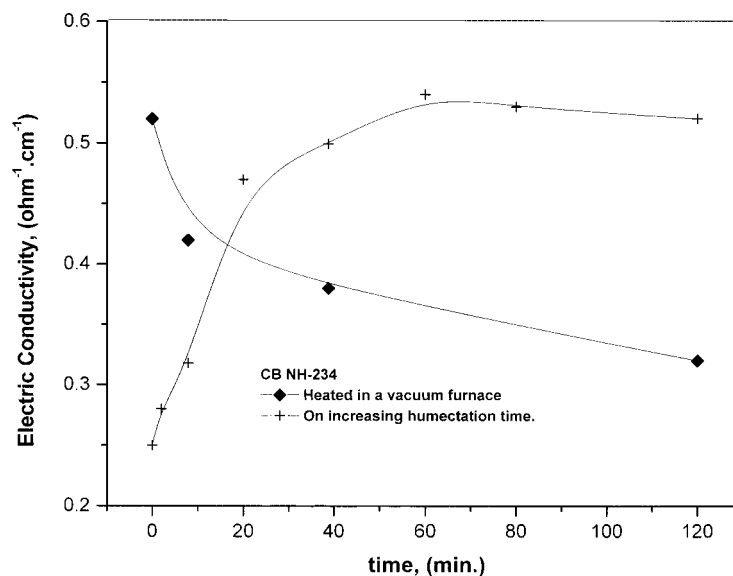


Figure 6 Influence of water on the electrical conductivity of carbon black agglomerates.

that CB agglomerates can be obtained with low electrical resistance and greater absorption capacity. The electrical conductivity of the FA resins were measured from 30 to $>400^{\circ}\text{C}$ and it was observed that the conductivity increased with the temperature.²⁷

Electrical and thermal conductivity of carbon black agglomerates

Figure 4 shows the dependency of electrical conductivity of CB agglomerates as a function of $r(\text{Q}/\text{NHUMO})$ for LSP, *nejayote*, and water. It was found that, for all systems (except when water was the additive), the electrical conductivity increases with the increase in $r(\text{Q}/\text{NHUMO})$, followed by a decrease. The maximum conductivity of $0.99 \Omega^{-1} \text{cm}^{-1}$ for LSP, and $0.76 \Omega^{-1} \text{cm}^{-1}$ for *nejayote* were attained at about $r(\text{Q}/\text{NHUMO}) = 3.5 \text{ mL/g}$. The electrical conductivity of CB agglomerates depends on the amount and type of additive. These results may be explained as follows. In the conductivity of CB agglomerates two mechanisms can be present: the formation of conductive networks of CB in the bulk material and the influence of the alkaline ions. For instance, K^+ favors ionic conductivity (see Fig. 5). The Ca^{2+} ions show only a slight influence on this mechanism of conduction because Ca^{2+} ions tend to form salts of low solubility, mainly CaCO_3 . Beyond a certain critical limit, this conduction decreases because of the fact that the CB and additive become interlinked, thus affecting the network structure of CB.²⁸ Thus the net result is a reduction in the number of conductive networks and, consequently, of the electrical conductivity.

Adsorbed water facilitates ionic conduction of K^+ (see Fig. 6). On increasing the wetting time of the samples CB-234 samples, the electrical conductivity increased until a saturation value of $0.52 \Omega^{-1} \text{cm}^{-1}$ was attained.

Besides, when the CB agglomerates with LSP were heated in a vacuum furnace, a reduction of conductivity to a value of $0.32 \Omega^{-1} \text{cm}^{-1}$ was observed. Many experimental data confirm the role of the residual solvent on the conductivity and other properties of polymers and polymeric composites.^{16,29,30} For example, the conductivity of polyaniline may change by three orders of magnitude depending on the amount of residual water retained in the polymer.³¹

As shown in Figure 3, the electrical conductivity of CB/FA composite increases with the increase of (FA/CB) volume ratio. These observations are consistent with the presence of open furan rings to form new unsaturated structures that increase the electrical conductivity.^{19,27,32,33} Investigation of the furan monomer-CB composites and the influence of temperature on their properties is now in progress.

Thermal analysis

The agglomerates with *nejayote*, LSP, and FA are decompose in a simple thermal step, as can be observed in Figure 7. In all cases, the thermal decomposition takes place in the temperature range of $430\text{--}625^{\circ}\text{C}$; however, the thermogravimetric analysis data indicate that the agglomerates with FA show the higher thermal stability. CB agglomerates prepared with LSP and with *nejayote* have a similar thermal behavior.

CONCLUSIONS

The thermal stability and the influence of *nejayote* and LSP on the cohesive strength of CB agglomerates are similar. This result opens a potential application to *nejayote* as a CB agglutinant. *Nejayote* is an inexpensive

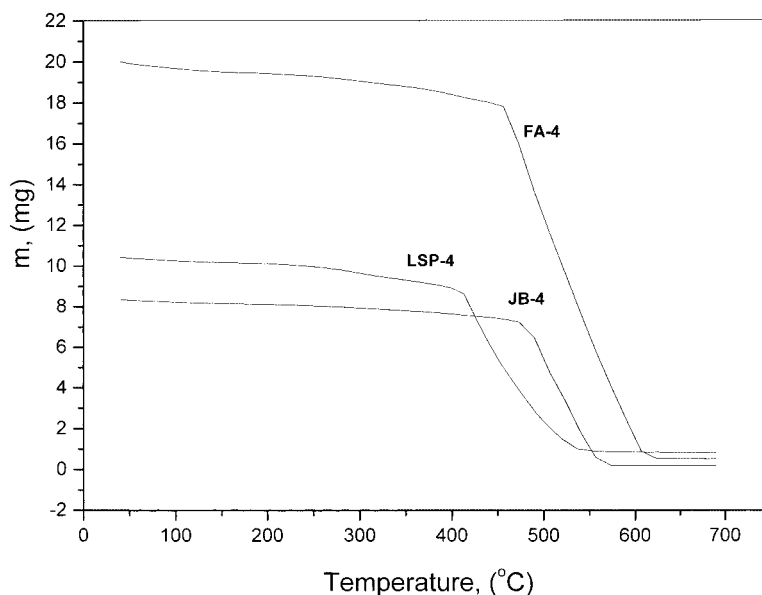


Figure 7 Thermogravimetric curves of carbon black agglomerates for potassium lignosulfonate, *nejayote*, and furfuryl alcohol as additives.

material and its use could help to solve a pollution problem in countries with industries based in the corn *nixtamalization* process. Also, FA can be used as an agglutinant. On the other hand, it was demonstrated that the influence of water is not significant. Electrical conductivity of the formed agglomerates depends on additive type and also on the additive volume used in their preparation.

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