Synthesis of a Plant-based Dust Suppressant and Testing on Coal from Moatize, Mozambique

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The dust suppressant was synthesized using *Pinus elliottii* resin as raw material for the new plant-based formulation. The dust suppressant formulation was prepared by solubilizing rosin in a ternary solution containing calcium chloride, ultrapure water, and ethanol in a molar ratio of 1:2:8. After centrifugation the supernatant was collected, 20 mL of 2 % γ-polyglutamic acid dissolved in an aqueous ethanol solution was added, and the mixture was stirred for 2 hours. The dust suppressant formulation included *Pinus* extract and γ-PGA acid, resulting in a plant-based dust suppressant. The performance of the new formulation in reducing PM_{10} was significantly superior (82.7 %) to that of water. This suppressant is suitable for spraying onto coal ore piles and hopper cars carrying coal ore. The wettability of the product was analyzed by the Walker test, which confirmed its adhesiveness to coal dust.

Keywords:
Pinus extract, γ-polyglutamic acid, coal, Moatize, particulate matter, dust suppressant

Introduction

Air pollution is a common situation in the mining sector, with its typical aspects of degradation of biotic and abiotic environments. Despite the remarkable advances and technological progress mankind has achieved, today we face local and global environmental issues resulting from the production and consumption patterns of industrial countries, which have led to environmental degradation, affecting nature's ability to provide the services and functions essential to life. The degradation of air quality is a major threat to human health. In 2013, 5.5 million premature deaths worldwide, one in every 10 deaths, were attributed to air pollution[1]. In 2015, the International Agency for Research on Cancer (IARC) considered airborne particulate matter (PM) one of the leading causes of cancer death as well as the main human carcinogen – classified in group 1 – (IARC, 2015). Human exposure to PM can strongly affect the pulmonary lobes and the heart, especially particles with an aerodynamic diameter of less than 0.1 micron, because they can reach the alveolar acini of the respiratory tract[2]. Energy is largely produced by burning fossil fuels, including coal, oil, and natural gas. Coal supplies 30 % of the world's primary energy needs and generates about 42 % of its electricity, and is also used in more than 70 % of the world's steel production[3]. Total world coal production has reached a record level of over 7,212 million tons[4]. Mozambique stands out in this scenario for its large coal reserves located in Moatize District, Tete Province. Large-scale exploration and exploitation of extractive natural resources in Mozambique could lead to irreversible negative environmental impacts. In operation since 2011, the Moatize coal mine produces coking and thermal coal, which is transported by rail to the port of Nacala. The value of known reserves in the Moatize coal deposit, which is considered one of the largest deposits in the world, is estimated at just over 2.5 billion tons.

Particulate matter (PM), total suspended particles (TSP), coarse inhalable particles (PM_{10}), fine inhalable particles ($PM_{2.5}$), and smoke are labeled as particulate matter. There is a group of air pollutants composed of dust, smoke, and all sorts of solid and liquid materials that remain airborne because of their small size. Coarse inhalable particles ($PM_{2.5–10}$) have an average aerodynamic diameter ranging from 2.5 to 10 μm, while fine inhalable particles ($PM_{2.5}$) are smaller than 2.5 μm[5]. The descriptions of air quality and seasonal variations in Moatize, Mozambique, reveal a high air pollution potential due to particulate matter.

Physicochemical methods to control particulate matter emissions involve the use of solutions of dust suppression products. These products, which

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are sprayed onto the surface of small particle size ore in the form of aqueous solution dispersions, effectively suppress dust over a long period of time by forming a protective film on the ore surface, adhering to it or even absorbing moisture from the air, and thus hindering the dispersive action of wind. Although the most common dust suppressant is water, it is well known that its large-scale application may be unfeasible because it evaporates rapidly, especially at air temperatures of around 50 ºC, which are common in Moatize. Mining companies typically choose the most economically feasible option to handle dust emission problems. Physicochemical dust suppressants are the cheapest option in the transportation of ore.

Water evaporation, in this case, can be potentiated by factors such as volatility, when the surface tension of water molecules holds them within the liquid. In the presence of high humidity, the force exerted by atmospheric pressure prevents water evaporation. On dry days, atmospheric pressure is lower and water molecules are released into the air more easily. Moreover, when wind sweeps over the surface of water, it takes up some molecules with it, thereby contributing to local evaporation. Another factor contributing to evaporation is the area of dispersion of water. The larger the area the higher the evaporation rate, as can be observed in the arrangement of stockpiles and hopper cars.

Two types of climate prevail in Moatize District: the BSw dry steppe type with dry winters in the southern part, and AW, the rainy tropical savannah type climate in the northern part of the district. These climates are characterized by two distinct seasons, the rainy and dry seasons. The annual averages of maximum and minimum temperatures are 32.5 and 20.5 ºC, in November and July, respectively, with heat waves that can reach up to 50 ºC. The average wind speed at Tete Weather Station, the site closest to the study area, is 6.6 km h^{-1}, with a speed of approximately 5.5 km h^{-1} from December to July, and the remaining months with higher wind speeds averaging 8.7 km h^{-1}. The weather is considered calm when wind speed is 1.0 km h^{-1} or less, blowing in no given direction.

The main objective of this work was to use *Pinus elliottii* var. *elliottii* as a raw material in the formulation of a dust suppressant, thus meeting environmental standards and following a growing global trend. The importance of coal for economic development is indisputable, but coal mining can have harmful environmental effects, including air pollution. In the coal mining process, total suspended particulate matter emissions occur during the use of explosives, and in the drilling, crushing, storage, and transport of coal ore. Mining companies use dust suppressants to create a film on ore piles, thereby preventing losses caused by wind and other weather phenomena during transport. The Moatize mine in Tete Province in Mozambique has an annual production capacity of eleven million tons of coal. The new dust suppressant formulation was tested on coal samples collected in Moatize District, located in Tete Province in central Mozambique, Africa. Concomitantly, the levels of particulate matter (PM_{10}) were measured in neighboring communities located within the area of the mining concession, one of the world's largest deposits of coal.

Material and methods

The dust suppressant was synthesized using *Pinus elliottii* var. *elliottii* resin as raw material for the new plant-based formulation. The resin was subjected to vacuum distillation to separate rosin and turpentine. Before producing the suppressant, the rosin was chemically modified (sulfitation) to decrease its viscosity, since the crude extract is extremely viscous. Sulfitation was performed using 1 part rosin, 1.5 parts water, 3 % sodium sulfite, and 10 M HCl until the material reached a pH of about 3.0. The mixture was kept under constant heating and stirring, and refluxed for a period of 90 minutes. The viscosity of the dust suppressant formulation was determined using an SV-10 Vibro Viscometer.

During the application of a dust suppressant, the interface between the coal and the film formed by the dust suppressant should be monitored carefully to evaluate the contact region. The Walker sink test and laser scanning confocal microscope (LSCM) were used to determine whether the dust suppressant was adsorbed by the coal dust matrix. Morphological information about the Moatize coal sample was obtained using an environmental scanning electron microscope (FEI Quanta 250 ESEM). A conventional scanning electron microscope (LEO 440i SEM) coupled to an energy dispersive X-ray spectrometer (EDS) was also used. The joint use of SEM-EDS is very advantageous in the analysis of geological samples.

The mineralogical composition of the raw coal was analyzed by X-ray diffraction (XRD), using a Rigaku MiniFlex diffractometer equipped with a CuKα radiation source (λ = 0.15406 nm), 30 kV voltage, and 15 mA current, in a 5–80º degree rotation on a 2θ scale, 0.02º step size, 0.6 s per step. The results were analyzed using Diffraction Plus software, integrated with JPCDS – International Center for Diffraction Data (ICDD), version 2001.

The coal was analyzed by thermal gravimetry in the range of 10 to 1000 ºC, applying a heating ramp of 10 ºC min^{-1} in a nitrogen atmosphere, using a model Q500 thermogravimetric/differential thermal analyzer (TG-DTA) from TA Instruments.

Trace elements in the raw coal sample and particulate matter were identified in a Spectro Arcos Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES).

The standard used here was γ-PGA in sodium salt form, a product of Shandong Freda Biotechnology, with γ-PGA of 30 %. The presence of γ-polyglutamic acid functional groups was investigated by means of infrared spectroscopy. The infrared spectra of the compounds embedded in potassium bromide were recorded by IR spectroscopy in the range of 400 – 4000 cm^{-1}, using a Perkin Elmer 16 PC FTIR spectrophotometer.

To ascertain if the dust suppressant was adsorbed onto the coal matrix (200 mesh), a sample of coal dust was placed on a silica slide, covered with a coverslip, and examined under a laser scanning confocal microscope (Zeiss LSM 510 Meta) equipped with four lasers, which enabled excitation of the sample at various wavelengths.

Results and discussion

The level of toxicity of PM to human health depends on parameters such as chemical composition and mainly on the aerodynamic particle diameter (pd)[6]. The aerodynamic diameters of particles strongly govern the laws of physics of pulmonary deposition, which is why the U.S. Environmental Protection Agency (EPA) classifies particles as follows: fine particles PM 2.5 have an aerodynamic diameter between 0 and 2.5 μm; coarse particles PM 10 have an aerodynamic diameter between 2.5 and 10 μm[7]. Fine particles with a diameter of less than 0.1 μm are called ultrafine PM 0.1, while those smaller than 0.05 μm are known as nanoparticles (PM 0.05)[6].

High-energy ball milling is a powder processing technique that is used in the production of homogeneous materials. Mineral coal was placed in a horizontal attritor mill with a stainless steel container and chromium steel balls, operating at a rotation of 800 rpm, where it was milled for 1:30 h. The untreated coal became powdered in the first 30 min in the attritor mill, but the material was analyzed by SEM only after the pre-established time, to examine the crushed particle size, as illustrated in Fig. 1. The scale in Fig. 1 indicates that the particles have irregular shapes and their dimensions were not at the nanoscale. In fact, the smallest particle size was in the range of 10 μm. The coal powder then underwent ball milling until it reached a nanometer particle size, as determined by the BET method, which revealed a particle diameter of 10 – 123 nm and density of 1.25 g cm^{-3}.

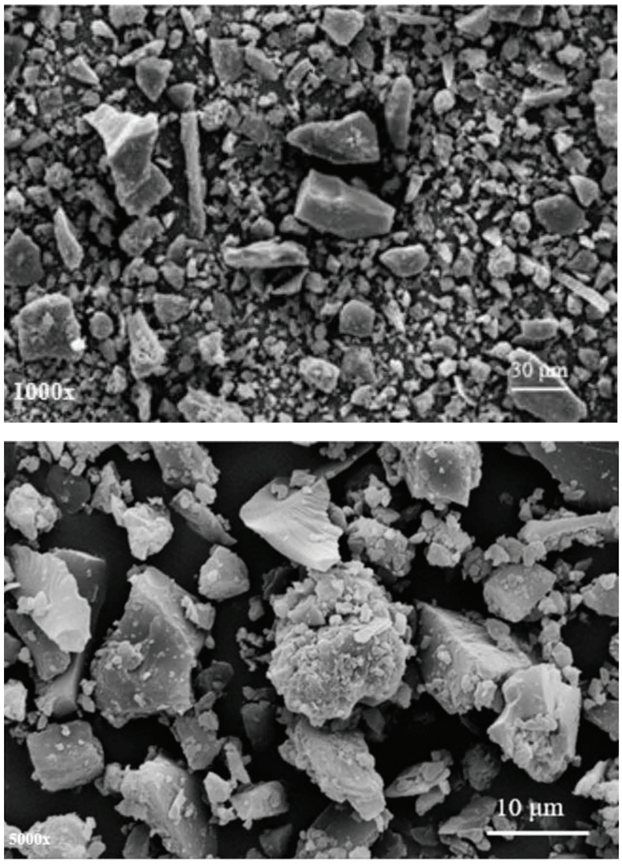


Fig. 1 – *SEM micrographs showing the external morphology of the raw coal (1000 and 5000x magnification)*

Water-absorbing dust suppressants whose composition includes calcium chloride stand out for their good hygroscopicity. Copeland *et al.*[8] stated that this hygroscopic reagent maximizes the efficiency of water as a suppressant of PM10 from iron ore, for example. Another compound with suppressive characteristics is natural polymers, particularly cellulose-based polymers. Starch is a material that has proved to be highly feasible, since it comes from a variety of sources, is low cost, its storage is stable and has excellent biodegradability[9,10]. Given the unfeasibility of using water, particulate control systems have been increasingly replaced by commercial dust-suppressing polymers, which maximize the efficiency and sustainability of the process. What must be kept in mind, however, is that each formulation is restricted to one type of mineral ore. In other words, the fact that there is a wide variety of suppressors in the literature and on the market does not mean that they can be used on every type of ore, due to environmental characteristics and the specificity of each mineral ore.

Coal from Moatize

The XRD diffractogram of the coal revealed the presence of quartz, clay minerals such as kaolinite, as well as iron oxides and oxyhydroxides such as hematite and goethite. The morphology of the raw coal (Fig. 1), which was examined by scanning electron microscopy (SEM), showed round and elongated particles ranging in size from 10 to 30 µm. Under 5000x magnification, the electron micrograph revealed the formation of large clusters of carbonaceous material with uneven surfaces.

The elemental analysis of Moatize coal showed high carbon content (78.7 %), with low contents of hydrogen (5.42 %), nitrogen (2.45 %), and sulfur (0.16 %). Lakshminarayana found hydrogen content ranging from 5 to 6 %, and carbon content ranging from 83 to 86 % in Moatize coal samples[11]. Manharage, in turn, identified carbon content varying from 78.71 to 86.71 %, hydrogen from 4.85 to 5.03 %, and nitrogen in the range of 1.87 to 2.13 %[12]. Particulate matter is a complex substance; therefore, it is not simply a question of determining its concentration but also of differentiating its diameter, analyzing its chemical composition, phases and morphology[13]. The chemical analysis of the particulate matter indicated the presence of sulfur (6431.19 mg kg^{-1}), manganese (68.73 mg kg^{-1}), and chromium (21.57 mg kg^{-1}), as well as 8.5 % silicon, 3.02 % aluminum, and 0.9 % iron. However, no arsenic was detected in the samples of particulate matter. A study on air pollution in a coal mining area in India showed concentrations of Fe> Cu> Zn> Mn> Pb> Cr> Cd> Ni. Environmental concentrations of iron found in particulate matter ranged from 1.43 to 28.48 g m^{-3} [14]. Fig. 2 shows SEM micrographs of the particulate matter under 500 and 2000x magnification. Fig. 3 depicts the daily concentrations of particulate matter in the 25 de Setembro neighborhood, whose primary pattern is represented by particles with an aerodynamic diameter of ≤ 10 µm, i.e., inhalable particulate matter (PM_{10}). According to Mozambique's Decree No. 67/2010 on the Regulation on Environmental Quality and Effluent Emission Standards, the maximum permissible concentration of particulate matter in 24 hours is 150 µg m^{-3}.

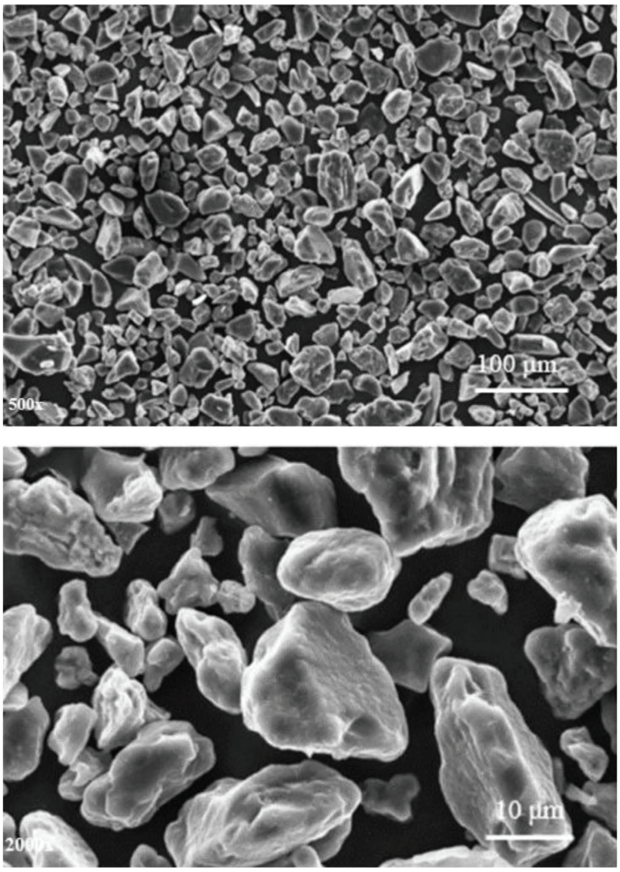


Fig. 2 – *SEM micrographs showing the external morphology of the particulate matter, PM_{10} (500 and 2000x magnification)*

TG-DTG curves measured at 10 ºC min^{-1} in a nitrogen atmosphere were analyzed to illustrate the thermal behavior of the Moatize coal sample (Fig. 4). The thermal decomposition behavior of coal takes place in three stages. The first stage (I) is associated with the loss of moisture of the material, which occurs at around 100 ºC. The second stage

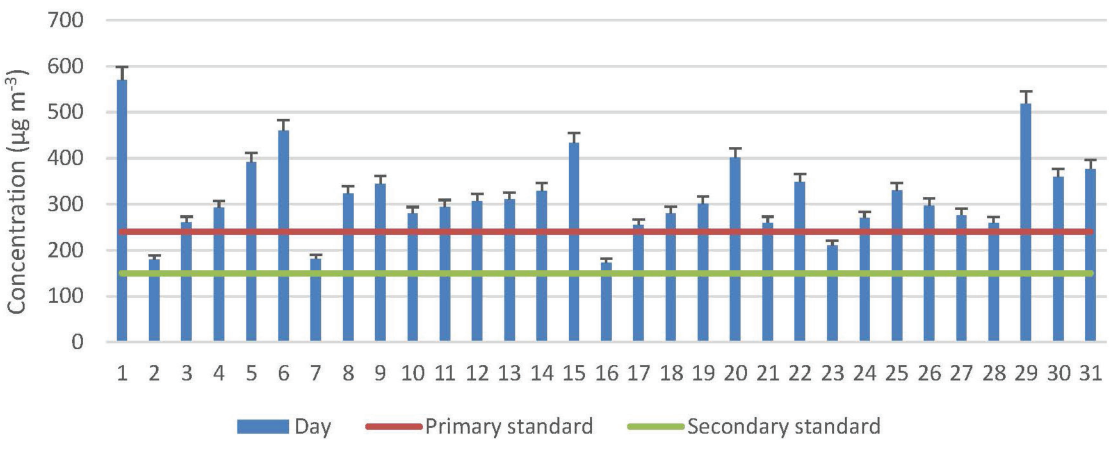
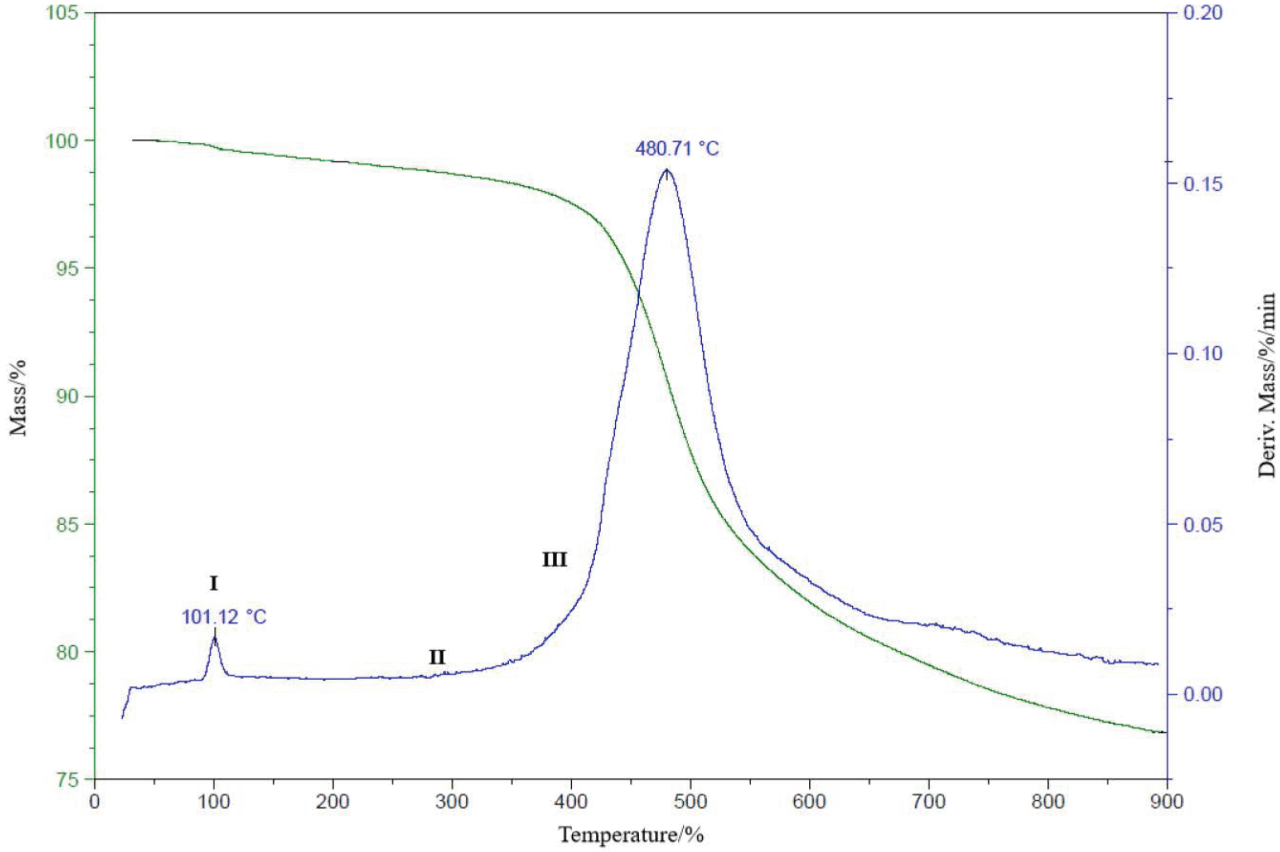


Fig. 3 – *Daily concentration of particulate matter (PM_{10}) in August 2015, in the neighborhood of 25 de Setembro*



F i g . 4 – *TG-DTG curves of Moatize coal measured at 10 °C min^{-1} in a nitrogen atmosphere*

(II) involves the material's primary combustion and loss of volatile matter. The third stage (III) involves the combustion of fixed carbon[15].

Synthesis of the dust suppressant

Coal dust is a naturally hydrophobic material that is difficult to suppress because it is not readily wettable. Surfactants have therefore been suggested as an effective reagent, since they enhance the wettability of fine coal particles[16–19]. However, many products contain toxic chemicals such as aromatic solvents. Polymers, which are also used in the formulation of dust suppressants, can form polymeric films on particulate matter.

The dust suppressant formulation was prepared by solubilizing rosin in a ternary solution containing calcium chloride, ultrapure water, and ethanol in a molar ratio of 1:2:8 ($CaCl_2$:C_2H_5OH:H_2O), with 10 % rosin under magnetic stirring at 350 rpm until complete dissolution at 65 ºC. The resulting solution was filtered under positive pressure using a retention membrane with 0.45 µm mesh openings. The filtered solution was then neutralized with 10 % sodium hydroxide, after which the suspension was poured into Falcon tubes, and centrifuged for 15 min (15000 rpm) at 24.6 ºC.

After centrifugation, the supernatant was collected, 20 mL of 2 % γ-polyglutamic acid dissolved in an aqueous ethanol solution was added, and the mixture was stirred for 2 hours. Gamma-polyglutamic acid (γ-PGA) is a water-soluble, biodegradable, biocompatible, and non-toxic anionic biopolymer obtained through fermentation from bacteria of the genus *Bacillus*. Biopolymers are of interest mainly because they offer an alternative for the substitution of synthetic polymers derived from petroleum, which pollute the environment, since they do not undergo biological degradation, i.e., they are non-biodegradable, and take decades to break down through chemical degradation[20–22]. This solution was kept in a water bath at 65 ºC under gentle stirring for 60 minutes, after which the pH was adjusted to 7.0. After cooling, a mixture of calcium chloride, sodium lauryl ether sulfate, and fluorinated surfactant (15:10:5) was added to the original solution and again placed in a water bath at 65 ºC under gentle stirring for 180 minutes. The apparent viscosity of the dust suppressant solution was 1.53 mPa s^{-1} at 23.3 ºC. The sample was diluted in ethanol to a concentration of 1.6 %.

The FTIR spectrum of the rosin sample shows an intense band at 3433 cm^{-1}, which is attributed to vibration of the O–H bond. Carboxylic acids are usually strongly associated through hydrogen bonds. Also visible in the spectrum are bands at 2869 cm^{-1} and 2934 cm^{-1} resulting from bending vibrations of

Table 1 – *FTIR absorption peaks (cm^{-1}) of rosin and γ-PGA 30 % sodium salt from Shandong Freda Biotechnology Co. Ltd., used as reference material, and of the dust suppressant produced in this study*

Sample	Amide I	Amide II	C=O	C–N	N–H	O–H
γ-PGA 30 %	1570.99	1450.74	1397.28	1278.37	687.64	3267.64
Dust suppressant	1693.32	1446.12	1458.18	1152.69	659.3	3387.4

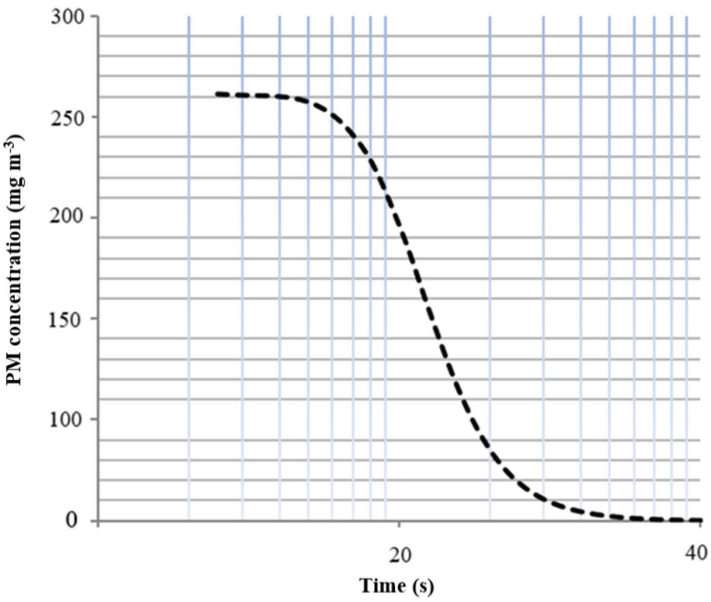


Fig. 5 – *Changes in dust concentration during a Drop Test, using the plant-based dust suppressant formulation*

the CH_3 group that is present in unsaturated hydrocarbons, such as resin acids. Note the intense band in the spectral region of 1695 cm^{-1}, resulting from bending of the C=O bond of the carboxylic group. The spectral region centered at 1385 cm^{-1} shows a band characteristic of the twisting vibration of CH_2 groups. These bands may be associated with the structure of resin acids. The stretching vibration at 968 cm^{-1} corresponds to the –COOH groups, while that at 1180 cm^{-1} corresponds to the aromatic groups.

The FTIR spectrum of the dust suppressant shows an intense band at 3280 cm^{-1} attributed to vibration of the O–H bond. The band at 876 cm^{-1} is attributed to a bending vibration of the C–C bond in aromatic rings. This type of vibration is caused by the dehydroabietic acid in rosin, which incorporates an aromatic ring similar to rosin due to the raw material of the product. The bands in the infrared spectrum indicate the presence of rosin and γ-PGA (see Table 1 and Fig. 3). The band in the IR spectrum at 1458.18 cm^{-1} corresponding to the dust suppressant containing γ-PGA is associated with C=O carbonyl group stretching vibrations, while the amide I band at 1693 cm^{-1} is caused by N–H bending vibrations. In addition, the spectral regions at 1152 cm^{-1} and 659 cm^{-1} show bands attributed to C–N stretching and N–H bending vibrations, respectively. The spectra of the dust suppressant samples containing rosin and γ-PGA exhibit bands from 3400 cm^{-1} to 3387 cm^{-1}, attributed to O–H bond stretching vibrations. The latter spectra are indicative of the presence of carboxylic, carbonyl, hydroxyl, and amide groups[23].

Walker sink test

The Walker Sink Test method was used to evaluate the ability of the suppressant to engulf fine particles. In this experiment, 100 mg of finely pulverized coal was released from a height of 253 cm onto the surface of the synthetic dust suppressant, which was deposited on the plate of an ultra-sensitive analytical balance. The product was tested to determine its ability to suppress inhalable particles, using a dust tower apparatus[16].

Fig. 5 shows how the dust concentration changed during the experiment in response to the use of the plant-based suppressant. The quantity of PM_{10} was measured using an air meter. The system started collecting data at time 0. The material was released at ten seconds, and the dust concentration peaked within 33 seconds thereafter. As air circulated through the dust tower, the concentration decreased until all the dust was removed. Dust suppression by the formulation occurred within 33 seconds after the coal dust was released.

Fig. 6 illustrates the effect of dust suppressants on the generation of coal PM_{10}. The plant-based dust suppressant significantly reduced PM_{10}, making it an interesting product for the mining sector. The new plant-based dust suppressant formulation was 82.7 % more effective than water in suppressing PM_{10}.

The images generated by laser scanning confocal microscopy revealed the adhesion surface formed by the suppressant film on the coal particles. The optimal excitation wavelength for recording the emission spectrum of the fluorescent compound was chosen as a function of its absorption spectrum, in the range of 440 to 485 nm. The images depicted in Fig. 7 were selected using ImageJ software, highlighting the film formed on the coal grains. These images enabled us to analyze the effect of the product and its mechanism of action on coal dust containing the layer of dust suppressant. The dust suppressant produced from rosin and

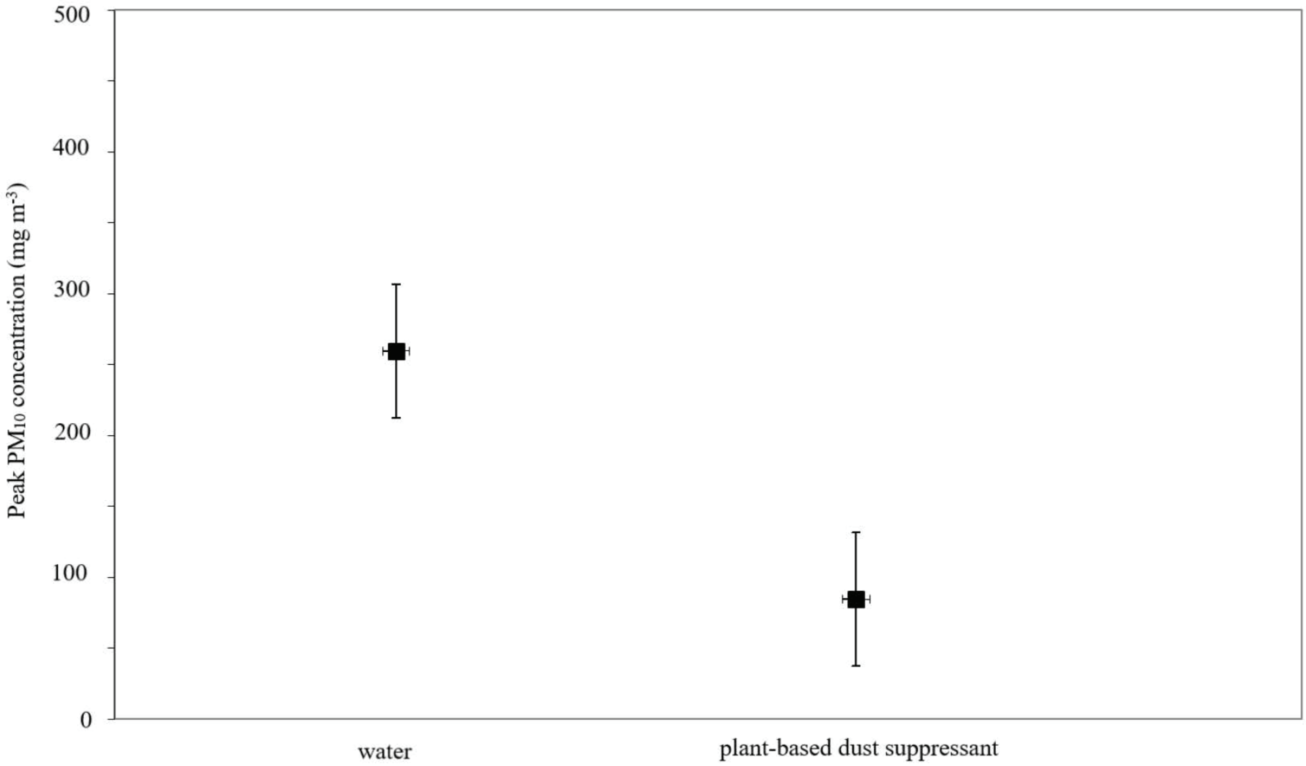


Fig. 6 – *Effects of water and dust suppressant formulation on the peak PM_{10} concentration (mg m^{-3})*

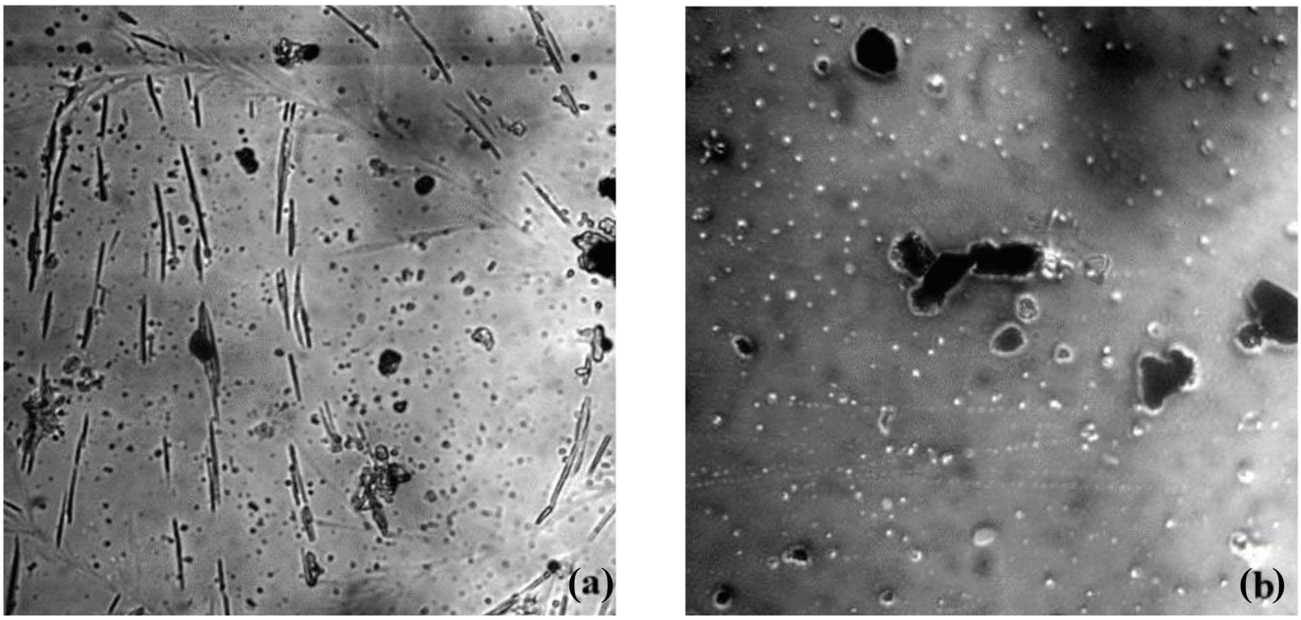


Fig. 7 – *Image recorded with a confocal microscope under: a) 100x, and b) 500x magnification, ISM 75 %. This technique was used to characterize the surface topography of the coal after spraying it with the plant-based dust suppressant.*

γ-PGA, which ensures high agglomeration and adhesiveness on dust particles, has a pH of 5.8 in aqueous solution and the appearance of a yellow viscous liquid. It was concluded that this product is suitable for spraying onto coal ore piles in coal storage yards or even as a solution for mining companies to spray onto coal in hopper cars to control the loss of fine materials during rail transportation.

Conclusion

Field data revealed high particulate matter concentrations in communities near the Moatize mine in Tete Province in Mozambique. The PM_{10} concentration in the neighborhood of 25 de Setembro reached 569 μg m^{-3} in August 2015. The dust suppressant produced from rosin is highly agglomerative and adhesive, with a pH of 5.8 and a yellow viscous liquid appearance. This suppressant is suitable for spraying onto coal ore piles and hopper cars carrying coal ore. The wettability of the product was analyzed by the Walker test, which confirmed its adhesiveness to coal dust. The effective suppression of PM_{10} is of interest to the mining industry, since they represent a problem from the standpoint of environmental legislation, and are harmful to public health. A wide range of suppressants to reduce environmental dust are available on the market, which use proven toxic surfactants and substances. The plant-based dust suppressant formulation was effective on coal, improving the wetting characteristics of the material. Coal dust is a naturally hydrophobic material, which is difficult to suppress since it does not wet easily. The performance of the new formulation was superior (more than 80 %) to that of water in reducing PM_{10}. In other words, water suppressants were much less effective than the plant-based dust suppressant. The genus *Pinus* is an abundant and low cost raw material with a chemical potential for the development of new products with high added value.

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