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## INFLUENCE OF PARTICLE SIZE ON THE EXPLOSIBILITY OF AIR CORK DUST MIXTURES

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Abstract. Flammability studies of air cork dust mixtures were conducted in the 20 L US Bureau of Mines explosibility test chamber, using 2500 J pyrotechnicignitors. The dust burned as air-dispersed dust clouds and the uniformity of the particles dispersion in the chamber was detected through optical probes. The range of tested particle sizes went from 45 to 350 mm, whereas the dust cloud concentration was up to 600-700 g/m<sup>3</sup>. Measured explosions parameters included minimum explosible concentration, maximum explosion pressures and maximum rate of pressure rise. The effect of dust particle size on flammability was evaluated and was found that minimum explosible concentrations were relatively independent of particle size below 180 mm. Highest explosion pressure and rate of pressure rise, were detected at the finest tested sizes. The measured flammability limits for this heterogeneous system are shown to be consistent with the established mechanisms and processes for homogeneous flame propagation, provided that the heating and devolatilization of the solid fuel is also considered. The limitations on the rates of devolatilization of the solid particles became rate controlling at high burning velocities, at high dustloadings and for large particle sizes.

Keywords. Dust explosion, air-cork dust mixtures, explosibility

#### 1. Introduction

Cork dust is a by-product of the cork industry and particularly of the cork stoppers industry. Being composed by small cellolosic particles, its composition is around 97 % of volatile mater and 3 % of water, it is prone to induce frequent accidents with loss of human lives and destruction of industrial facilities, a common situation in the northern region of Portugal. Strangely, studies on the characterization of explosion conditions of this dust are rare.

An explosion can be defined as a propagation of a flame in a pre-mixture of suspended dust in a gaseous oxidant such as air, in a closed vessel. During the explosion there is a rapid and abrupt energy release which produces a pressure wave. The resulting rapid oxidation of the fuel dust leads to a rapid increase in temperature and pressure. The combustion properties of a dust depends on its chemical and physical characteristics, specially on the particle size distribution (Eckhoff, 1997).

For a two phase mixture of a dust in air, the perfect mixture is impossible, and at least one additional variable is required in order to define composition on the macroscopic scale, which determines its combustion behavior. That additional variable is the particle diameter. In other words, a given amount of solid material, coal or cork for instance, suspended in air inside a container presents no explosion hazard; however, the same amount when pulverized into a fine dust of less than 100  $\mu$ m in diameter and uniformly dispersed in air, inside that same volume, may become an explosive mixture. The thermodynamic states of those two systems are essentially identical, but their dynamic behavior is vastly different. Thus, the state of the heterogeneous system is not uniquely defined by its macroscopic composition, temperature and pressure, the particle diameter is critical in defining the fuel concentration on the microscopic scale that determines its dynamic combustion behavior (Hertzberg, 1986).

This paper concerns a small part of the study and definition of explosion characteristics of air cork dust mixtures and is oriented towards the analysis of the influence of particle median diameter on the minimum explosion concentration, on the maximum explosion pressure and on the maximum rate of pressure rise, for dust explosion at initial normal pressure and temperature.

#### 2. Experimental Set-up

Cork dust explosibility results discussed in this paper were obtained in a near-spherical 22.7 L test chamber, as shown in Fig. (1). This chamber is made of stainless steel and is a standard laboratory test chamber (ASTM E1515; ASTM E1226), used extensively for dust explosions studies of carbonaceous and elemental dusts (Hertzberg,1986; Cashdollar, 1988; Cashdollar, 1994). Its dimensional charateristics were provided by the Pittsburg Research Laboratory formely parte of the US Bureau of Mines (USBM) and now at the National Institute for Occpational Safety and Health (NIOSH).



Figure 1. Test chamber of 22.7 L.

Two optical dust probes (Conti, 1982; Cashdollar, 1981) were used to evaluate the uniformity of the dust dispersion by measuring the light transmission over a 38 mm path length through the dust cloud. Thin jets of air keep the windows of the probe clean.

The evolution of the explosion pressure and rate of pressure rise during the explosion was measured with a pressure transducer with internal diaphragm for absolute pressures in the range of 0-10 bar. This is an IMT, model 3245, with an accuracy of 0.5 % (F.S.), reproducibility  $\leq \pm 0.05\%$  (F.S.) and a response time  $\leq 1$  ms (within 10 to 90 % of F.S.).

The data acquisition system is composed by a personal computer of 66 MHz inside of which is installed a data acquisition board from KEITHLEY-Metrabyte, model DAS-1601. This board is a multifunction, high speed, analog and digital interface, that offers 8 differential or 16 single-ended analog inputs with 12-bits resolution at 100 ksamples/s. Two output channels of 12-bits for digital to analog converter are also provided. The experimental dust concentration reported for the chamber is the mass of dust divided by the chamber volume.

#### 2.1. Test Procedures

After the dust charge and the ignition device have been placed in the chamber, the top lid is bolted on and the chamber is partially evacuated to an absolute pressure of 0.20 bar. Then, a short blast of dry air from a 16 L auxiliary dispersing tank with a 0.3 seconds duration, disperses the dust and rises the chamber pressure to about 1 bar. The ignitor is activated after an additional delay of 0.1 s resulting in a total ignition delay of 0.4 s, from the start of dispersion until ignition, as suggested by the standard test procedure (ASTM E1515; ASTM E1226). The ignition sources used were electrically activated pyrotechnic ignitors composed of 40% zirconium, 30% barium nitrate and 30% barium peroxide and were supplied by Fr. Sobbe, from Germany. They delivered their energy in about 10 ms and when ignited they produce a dense cloud of very hot particles and little gas (Hertzberg, 1986). The 2500 J ignitors by themselves produce a pressure rise of about 0.27 bar in the chamber, as verified in calibration and set up experiments.

An example of the evolution of the absolute pressure and rate of pressure rise during the explosion of 300 g/m<sup>3</sup> of cork dust of 66  $\mu$ m particle diameter is shown in Fig. (2). The pressure trace in Fig. (2) starts at the partially evacuated value of 0.24 bar. The blast of air that disperses the dust starts at the time of 0.72 s and has a duration of 0.3 s, rising the inside pressure up to 0.95 bar. At this pressure the ignitor is activated 0.1 s later, guaranteeing that the dispersing

process has finished. The maximum explosion pressure, determined from the peak of pressure-time trace, is about 7 bar, and the rate of pressure rise corresponding to the steepest slope of the rising portion of the trace and is about 160 bar/s.





#### 3. Results

In order to study the overall explosibility characteristics of cork dust mixtures, tests were made over a range of concentrations that went up to 600-700 g/m<sup>3</sup> for six different particle diameters. Experiments were carried out with cork particles of 45  $\mu$ m, 66  $\mu$ m, 106  $\mu$ m, 180  $\mu$ m, 274  $\mu$ m, and 324  $\mu$ m particle diameters. Particles were screened with ASTM sieves and the particle sizes referred above are the arithmetic mean of mesh sizes of the two consecutive sieves, containing the particles.

From the knowledge of the maximum explosion pressure and rate of pressure rise obtained for each tested particle size Fig. (2), curves of the evolution of these parameters against dust concentration can be obtained Fig. (3).

#### 3.1. Efect of dust concentration.

A summary of the evolution of the pressure explosion and rate of pressure rise with the dust concentration obtained for particles of 66  $\mu$ m and 274  $\mu$ m diameter is shown in Fig. (3). Identical traces of the evolution of explosibility parameters as function of dust concentration, have been obtained for the other particle diameters.

In the upper part of the figure, the pressure ratio *PR* is the maximum absolute explosion pressure (with the pressure rise duo to the ignitor subtracted) divided by the pressure at ignition. In the lower part  $(dP/dt)V^{d/3}$  is the size normalized maximum rate of pressure rise for each explosion test (ASTM E1515).

The data in the figures show that below certain dust concentrations, explosions are not observed. This is the minimum explosible concentration. The criteria for significant flame propagation were the same as used previously at

USBM (Hertzberg, 1986; Chasdollar, 1992; ASTM E1515). These criteria are that the maximum explosion pressure  $PR^{3}2$  bar and that the volume normalized rate of pressure rise  $(dP/dt)V^{l/3} = 1.5$  bar.m/s.

For the examples in the figure, the obtained values for the minimum explosion concentration are 40 and 50 g/m<sup>3</sup>, respectively for 66 and 274  $\mu$ m particles.

At higher dust concentrations, the maximum pressure and rate of pressure rise level off as all of the oxygen in the chamber is consumed, but there is no evidence of a rich limit for the dust as is normally observed for hydrocarbon gases (Cashdollar, 1996).



Figure 3. Evolution of maximum explosion pressure and rate of pressure rise with dust concentration for cork dust air mixtures of 66 µm and 274 µm particle diameter

One explanation of this effect is that the solid phase fuel must first devolatilize before it can mix with the air. As soon as sufficient volatile are generated to form a stoichiometric concentration in air, the flame front propagates rapidly through the mixture, before excess volatiles can be generated (Hertzberg, 1986b). From Figure (3) it can be seen that the maximum values of explosion pressure are 7.2 and 6 bar and the maximum rate of pressure rise are 52 and 12 bar.m/s, for 66 and 274  $\mu$ m particles.

#### 3.2. Efect of particle size.

Data showing the effect of particle size on the minimum explosion concentration,  $C_{min}$ , maximum explosion pressure,  $P_{max}$ , and maximum rate of pressure rise,  $(dP/dt)_{max}V^{1/3}$ , of cork dust are shown in Fig. (4) and Fig. (5).

The initial flat region in Figure (4) shows that for fine particles the minimum explosion concentration is relatively independent of particle size. As particles become coarser, size dependence appears above some characteristic diameter. That characteristic diameter for cork dust is near 180  $\mu$ m. The minimum explosion concentration then increases rapidly with diameter, until a critical diameter is reached above which the coarse dust is essentially no explosive. The minimum explosive concentration obtained in Fig. (4) for cork dust is 41 g/m<sup>3</sup>.



Figure 4. Effect of mean particle diameter on minimum explosion concentration of cork dust air mixtures.

These results are most readily explained in terms of the following sequence of processes for flame propagation in the dust air mixtures proposed for some investigators (Hertzberg, 1982; Hertzberg, 1990): first, the heating and devolatilization of dust particles as they approach and enter the flame front; secondly, the mixing of emitted volatile with air in the space between particles; and thirdly, the gas phase combustion of the resultant fuel-air mixture. The minimum explosion concentration curve as function of particle diameter is flat below the characteristic diameter, because for lean concentrations near the flammability limit the burning velocity  $S_u$ , is small and the depth of penetration of the devolatilization wave exceeds the particle radius which means that the particle devolatilizes completely within the flame front (Hertzberg, 1986). Thus, it is the total combustible volatile yield of the finer dust particles, and the reaction of the devolatilized hydrocarbons burning homogeneously in the gas phase that determines the minimum explosion concentration. The curve begins to turn upward at the characteristic diameter because the particles become too coarse for the devolatilization process to be completed within the flame front and there is a transition to devolatilization rate control. For the coarser particles only the surface regions can devolatilize within the flame front, and a larger mass loading of dust is required to generate a lean limit concentration of combustible volatile. As the critical diameter is approached asymptotically, the devolatilization process becomes dominant, and the rate of evolution of volatile for the coarser particles is just too slow to sustain flame propagation (Hertzberg, 1990).

In Figure (5) the two curves show that the maximum pressure and rate of pressure rise are found at the smallest tested size and have the maximum values of 7.2 bar and 52 bar.m/s respectively. Then, with increasing particle size, the pressure values decline slowly, whereas the rate of pressure rise decrease is stronger.

The tendency of the maximum explosion pressure curve with increase of particle diameter is an indication that there are not significant changes in the volatile concentration of gas phase. At elevated concentrations, at which are obtained the maximum explosion pressure and the maximum rate of pressure rise, the burning velocity is high but the depth of penetration of the combustion wave, which varies inversely with the burning velocity  $S_u$ , decreases and becomes less than the average particle radius (Chasdollar, 1985; Smoot, 1976). The excess undevolatilized fraction mass of cork plus

the residual char, at the higher dust loading, makes no contribution to the flame propagation process; it begins to absorb a large fraction of the flames heating flux quantified by  $kS_uc_p \mathbf{r}(T_b-T_u)$  (Hertzberg,1982) and the effective heating flux, that drives the fresh pyrolysis wave ahead of the flame front, is diminished. As the particle diameter increases still further, the pyrolysis wave progresses more slowly into each particle, and this effect is accentuated with the increases of particle diameter. As a result, there is a smaller contribution of volatile from each particle that is compensated for by the higher surface area of the particles.



Figure 5. Effect of particle diameter on maximum explosion pressure and maximum rate of pressure rise of cork dust air mixtures.

The strong dependency of the maximum rate of pressure rise relatively to the particle diameter is caused by the accentuated diminution on the velocity of the devolatilization process, when the particle diameter increases. In fact the velocity of the combustion reaction is strongly dependent on the rate at witch the volatiles are generated, and these are retarded with the decreasing of the progress of the devolatilization wave through the particles. All this process will be reflected in the accentuated decrease of the burning velocity  $S_u$  and as maximum rate of pressure rise is proportional to  $S_u$ , they diminish drastically with the increase of the particle diameter, as is shown by several models (Bartknecht, 1981; Nagy, 1983)

Figure (6) shows a scanning electron microscope (SEM) photomicrograph of a cork dust particles of 66  $\mu$ m diameter. Examples of (SEM) photomicrographs of the same particle after explosions at dust cloud concentrations of 160 g/m<sup>3</sup> and 220 g/m<sup>3</sup> are shown in Figs. (7) and (8).



Figure 6. Scanning electron microscope (SEM) photomicrographs of an cork dust particles of 66 µm diameter

These figures present the morphological changes resulting from dust explosions in the 22.7 L chamber, and as can be seen the structure of the surface of the residue are strongly altered compared to the unburned cork particle Fig. (6). Same particles form cenospheres, as clearly observed in Fig. (7), and the volatiles are emitted through the holes seen in the residues, Fig. (8).



Figure 7. Scanning electron microscope (SEM) photomicrographs of an cork dust particles of 66  $\mu$ m diameter, after explosions at dust cloud concentrations of 160 g/m<sup>3</sup>.



Figure 7. Scanning electron microscope (SEM) photomicrographs of an cork dust particles of 66 μm diameter, after explosions at dust cloud concentrations of 220 g/m<sup>3</sup>.

The photomicrographs in Fig.(7) and Fig.(8) provide evidence that the pyrolysis process proceeds in the form of a devolatilization wave front that is initiated at the exposed surface and then propagates through the particle.

#### 4. Conclusions

Particle size has an important effect on the explosibility of cork dust. The results obtained show that the finest particles produce more violent explosions than the larger ones, and the evolution of the explosion parameters with the particle diameter show that the explosion pressure is essential a thermodynamic component while the maximum rate of pressure rise has a strong kinetic component.

The explosion of cork dust follows the propagation flame mechanism proposed by Cashdollar and Hertzberg. In these theories, the flame propagation in the gaseous phase combustion is preceded by the devolatilization process of the dust particles. For the smaller particles the combustion process is controlled by the combustion velocity, whereas for the bigger particles the combustion process is controlled by the devolatilization rate.

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