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Influence of Operating Conditions on Pre-wetted Powder Explosion

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Pre-wetting of powders or wet granulation are commonly used in industrial processes and the presence of a flammable liquid compound can affect the ignition sensitivity and explosion severity of such admixtures. This study shows that for risk assessment, it is not possible to consider this type of admixture to be equivalent to a hybrid gas/solid mixture of the same composition. Indeed, if the introduction of a solvent tends to increase the explosion risk, pre-wetting also significantly modifies the particle size distribution of the admixture, which counterbalances the positive contribution of the flammable liquid. Dispersing the dust requires then a greater dispersion pressure to reach the same surface area between solid fuel and oxidizer. In the case of the determination of the minimum ignition temperature, such changes lead to a modification of the optimal operating conditions. It should also be noted that the fuel equivalence ratio is a good indicator for assessing the chemical contribution of such admixtures.

1. Introduction

Because of their frequent occurrence in industry, hybrid mixtures have been the subject of in-depth studies of their risk of explosion in recent years. However, while atmospheres containing both combustible gases or vapours and powders have been regularly addressed (Gabel and Krause, 2019; Heilmann et al., 2024, Wu et al., 2023), the particular case of powders prewetted by a liquid has received little attention (Hossain et al., 2014). There are many industrial applications where this configuration needs to be considered, such as the wet granulation of pharmaceutical or food powders, or the hexane extraction of oil from oleaginous seeds, treating soil contaminated with hydrocarbons (Dufaud et al., 2022) or adding essential oils to powdered animal feed. The case of solid adsorbents that have accumulated volatile organic compounds can be related to this topic, although the quantities are smaller and the initial state of the solvent is gaseous.
A previous study carried out by Hossain et al. (2014) demonstrated a global explosivity enhancement when the powder was pre-wetted. This evolution depended on the fuels nature (solid and liquid) and on the method of admixture. A slight decrease of the maximum explosion pressure was notably observed for microcrystalline cellulose/solvents mixtures, but the phenomenological reason for these developments, whether negative or positive, has not been developed. Moreover, tests were only performed with a fixed solvent concentration (set at 80 % of the lower explosive limit) and the impact of solvent concentration still needs to be assessed. Indeed, the addition of solvent can play several contradictory roles: i) it provides a fuel that is generally easier to vaporise than the powder, thus helping to reach the LEL and reducing the diffusional limitation of oxygen, ii) because of its enthalpy of vaporisation, it acts as a heat sink during the ignition phase, iii) it modifies the morphology of the powder, requiring more energy to disperse it and iv) limiting the accessibility of oxygen to the surface of the agglomerates before their breakage. Given the small number of studies carried out on these mixtures, several mixtures of powders (e.g. cellulose, carbon black) pre-wetted with solvents (e.g. ethanol, water) were tested in this work. Two mixing methods were tested: dry powder dispersed in air containing solvent vapour, or powder mixed directly with the solvent before its dispersion. Both explosion severity and Minimum Ignition Temperature (MIT) were determined. The aim of this work is to determine the optimum conditions for testing this type of

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admixture to obtain the most relevant results from an industrial point of view or the most conservative results from a process safety perspective.

2. Materials and methods

Although this study was based on theoretical considerations, the mixtures considered were chosen to be consistent with actual applications in the pharmaceutical, paint or waste treament industries.

2.1 Powders, solvent and granulation method

Three powders were chosen with regard to their different particle size and physical interactions with the solvents: cellulose (Avicel PH-101), carbon black and a sample of soil extracted from a polluted site (Dufaud et al., 2022). The characteristic diameters of their particle size distribution (PSD) were determined ex-situ by dry measurement (Malvern Mastersizer 3000 - Aero S dry dispersion unit 2-bar) and are presented in Table 1. Ethanol, diesel and water were used as solvent. It should be highlighted that some microcrystalline cellulose/ethanol mixtures were already studied by Hossain et al. (2014), which will give a comparison basis.

Table 1: Characteristic diameters of the particle size distribution of the powders

Diameters / Powder	$d_{10,ini}$ (µm)	$d_{50,ini}$ (µm)	$d_{90,ini}$ (µm)
Cellulose PH-101	21	59	140
Carbon black	3	9	64
Polluted soil	81	225	472

Admixtures were prepared by sprayed fluidized-bed granulation (Singh et al., 2022). In order to ensure the homogeneity of the samples, they were mixed during one hour in a chaotic stirrer (Turbula) before testing. Figure 1 shows the morphological evolution of the pre-wetted powders (cellulose/ethanol) as a function of the solvent content. If slight changes are noticed from 0 to 5 vol%, no agglomerates or macroscopic granules are visible. However, cohesive interactions are clearly observed for solvent contents greater than 10 vol%.

Figure 1: Macroscopic observation of particle size changes due to granulation (Avicel PH-101/Ethanol)

2.2 Explosion severity and minimum ignition temperature

The explosion severity of the raw products and admixtures was determined using a 20L sphere, according to ISO/IEC 80079-20-2, 2016. Two methods were used to inject the fuels: i) the pre-wetted powder is directly put in the dust canister and injected through the dispersion nozzle in pure air (20 barg), or ii) the dry powder is injected in the sphere (20 barg) where a certain amount of solvent has previously been vaporized in air to reach 0.4 barg (Heilmann et al., 2024). Two 5kJ chemical igniters were used as ignition source, the ignition delay time was set at 60 ms and pre-ignition pressure rise at 0.64 ± 0.02 bar. Most of the experiments were carried out using a rebound nozzle, but a standardized mushroom nozzle was also used for a few tests to place the powder directly in the sphere, avoiding its fragmentation due to shear stresses. The minimum ignition energy (MIT) of pre-wetted admixtures was determined using a Godbert-Greenwald oven, according to ISO/IEC 80079-20-2. The dispersion pressure was varied over a 0.3 to 0.5 barg range and the mass introduced was 0.2 to 0.5 g.

2.3 In-situ particle size measurement

As the standard 20L sphere does not allow the visualization of the dust dispersion, a 20 L sphere provided with five windows was used to record the evolution of the PSD during the injection of the admixtures. Measurements, from 1 to 875 µm, were carried out with a laser diffraction sensor equipped with R5 lens (Helos - Sympatec).

The same diffraction sensor was used to measure the PSD at the exit of the Godbert-Greenwald oven, as a function of the dispersion pressure and solvent content.

3. Results and discussion

3.1 Explosion severity: influence of the solvent content and mixing method

Figure 2 shows the results obtained for cellulose/ethanol mixtures, for two different mixing modes: i) pre-wetted powder (purple crosses) and ii) dry powder mixed with ethanol vapour (yellow triangles). It appears that, from 0 to 7 wt.% ethanol, the explosion severity of both kinds of mixtures is the same. Around 8.5 wt.% ethanol, an increase of both the maximum explosion pressure P_m and maximum rate of pressure rise dP/dt_m is noticeable, especially for the prewetted powder. From 10 to 30 wt.% ethanol, the explosion severity of the pre-wetted admixture is significantly reduced with regard to that of the dry dust dispersed in the vapour/air atmosphere. This trend is especially visible for the maximum rate of pressure rise, due to the marked decrease of dP/dt_m for the pre-wetted powder. It should be noted that such evolution is consistent with the morphological changes observed in Figure 1 at high solvent contents. A few tests were performed by placing the pre-wetted cellulose directly within the sphere in a standard mushroom nozzle. Even for the pure dust, both P_m and dP/dt_m were lower than those obtained using the rebound nozzle and injecting the dust through the pressurised canister. If in-situ PSD measurements confirmed that the powders are much more fragmented with the rebound nozzle, which suggests that the size-increasing effect of granulation is not as great as expected when the powder is injected into the sphere, the sole difference oxygen accessibility cannot explain the P_m reduction. The heat sink effect due to solvent vaporisation for large granules could potentially explain the reduction in combustion efficiency.

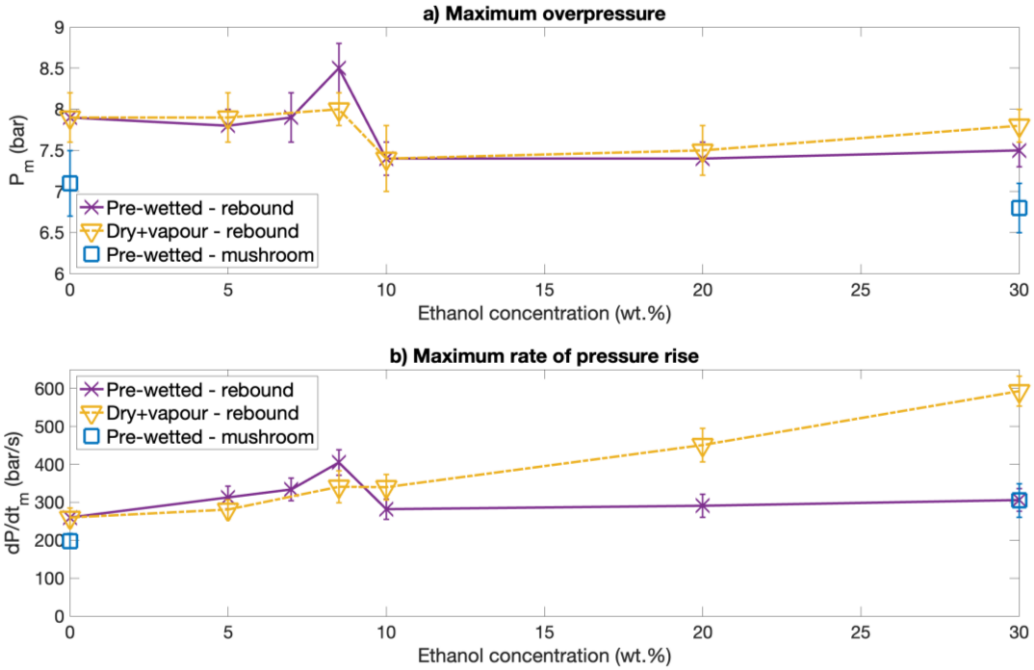

Figure 2: Effect of ethanol content, mixing modes and nozzles on the a) maximum pressure P_m and b) maximum rate of pressure rise dP/dt_m of Avicel PH-101/ethanol mixtures. Constant fuel moles: 0.062 mol.

Maximum values of P_m and dP/dt_m are 8.5 bar and 405 bar.s^{-1}, respectively, which corresponds to a Kst of 110 bar.m.s^{-1}. These results are consistent with the values obtained by Hossain et al. (2014), i.e. 7.8 bar and and 117 bar.m.s^{-1}. It should be stressed that the experiments were performed by keeping the number of fuel moles constant at 0.062 mol, considering both powder and solvent contribution. However, another approach is possible, as the stoichiometric air fuel ratio differ from cellulose to ethanol.

3.2 Explosion severity: tests at constant fuel equivalence ratio

Similar experiments were carried out, this time by keeping the fuel equivalence ratio (FER) constant (Lin et al., 2021), therefore leading to the increase of the total number of fuel moles when increasing the solvent concentration (Figure 3). FER was set at 2.25, corresponding to a cellulose mass of 10 g.

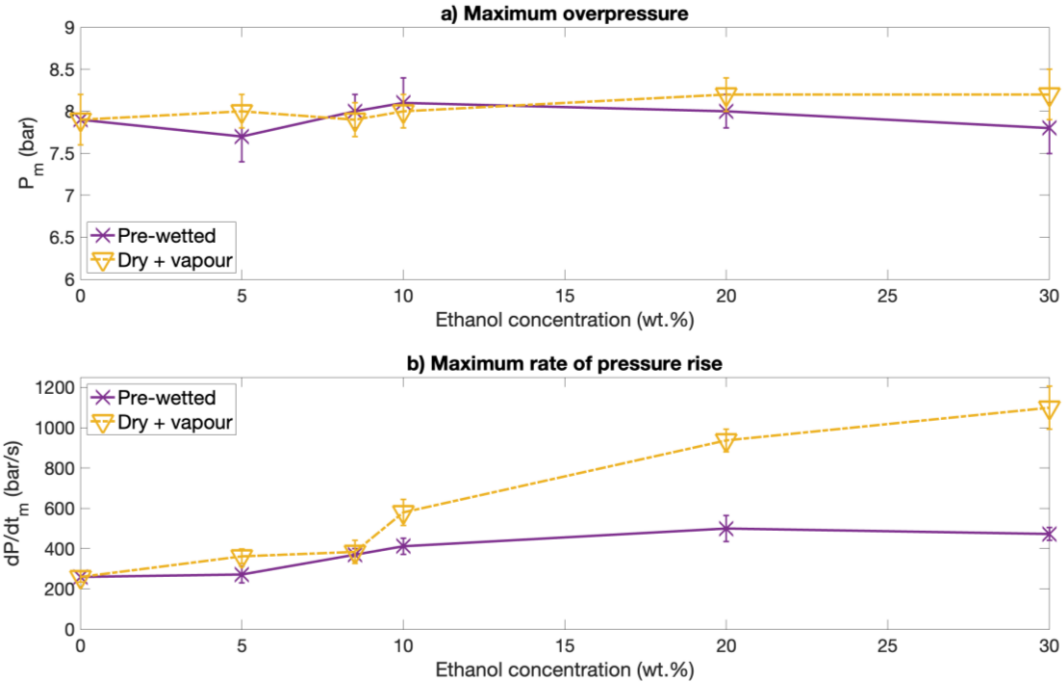

Figure 3: *Effect of ethanol content, mixing modes and nozzles on the a) maximum pressure and b) maximum rate of pressure rise of Avicel PH-101/ethanol mixtures. Constant fuel equivalence ratio: 2.25*

What is immediately noticeable in Figure 3a is that the maximum pressure varies very little as a function of the solvent content, whether it is introduced in the powder or in gaseous phase, which suggests that keeping the fuel equivalence ratio constant will enable a better assessment of the solvent role. Once again, the difference between the two mixing methods is not significant at low solvent concentrations. However, from 8.5 wt.% ethanol, the maximum rate of pressure rise of dry powders dispersed in an atmosphere enriched with ethanol vapor is considerably higher than that of the pre-wetted powder containing the same amount of solvent (Figure 3b). Indeed, for a mixture of 70 wt.% cellulose/30 wt.% ethanol, dP/dt_m varies from 473 to 1099 $bar.s^{-1}$ as a function of the mixing mode.

A substantial increase of the admixtures' explosivity is then observed when the solvent content increases but, at high solvent concentrations, this positive effect is counterbalanced by an increase in PSD. Such particle-size evolution not only modifies the energy required to disperse the powder, but can also, during the vaporization of the liquid bridge, create particle-particle interactions modifying their reactivity.

A similar approach was followed to determine the explosion severity of carbon black/ethanol admixtures at constant fuel equivalence ratio (FER = 1.25). Figure 4 shows a completely different behavior than that observed previously for cellulose. Although a slight increase in explosion severity noted between 5 and 20 wt.% solvent for vapor injection, this benefit is no longer visible at high concentrations. The magnitude of the effects is therefore highly dependent on the powder-solvent combination and on their interactions.

3.3 Minimum Ignition Temperature: influence of the solvent content and nature

Results in Table 2 show that the MIT decreases globally with the addition of solvent. Nevertheless, the variations depend greatly on the nature of the compounds and the liquid concentration. Lower solvent contents (2 to 8 wt%) were also tested leading to less significant decrease of the MIT.

Table 2: Minimum ignition temperature (°C) of prewetted powders

Solvent wt% / Powder/solvent	+ 0 wt%	+ 25 wt% solvent	+30wt% solvent	+ 30 wt% water
Cellulose / ethanol	440	430	400	460
Carbon black / ethanol	690	450	420	> 750
Polluted soil / diesel	> 1000	600	610	> 750

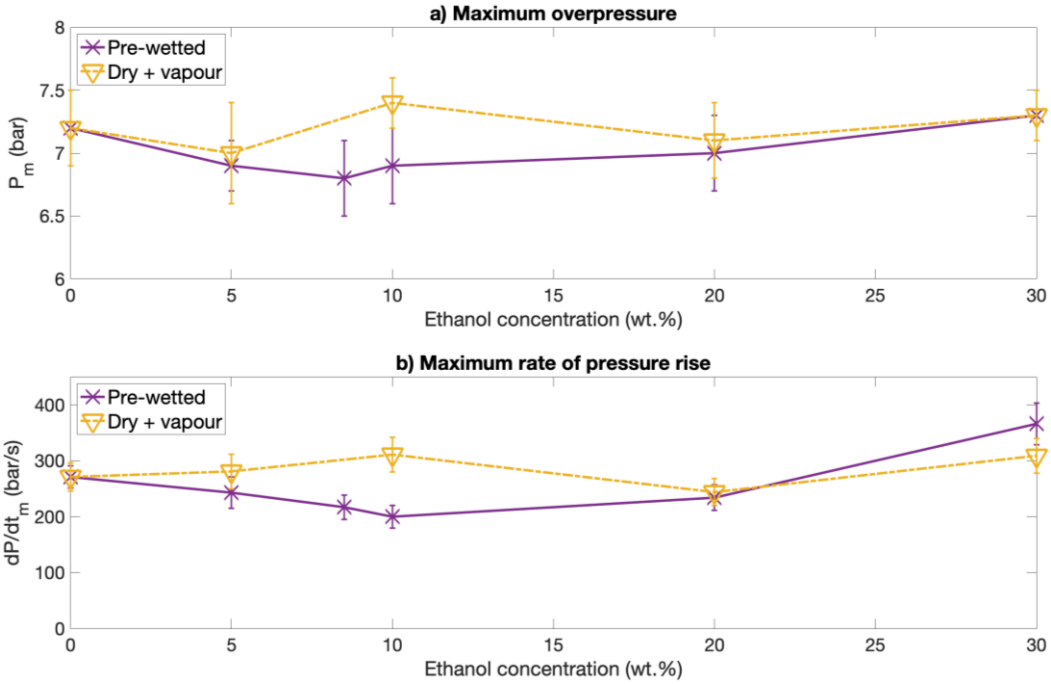

Figure 4: Effect of ethanol content and mixing modes on the a) maximum pressure and b) maximum rate of pressure rise of carbon black/ethanol mixtures. Constant fuel equivalence ratio: 1.25

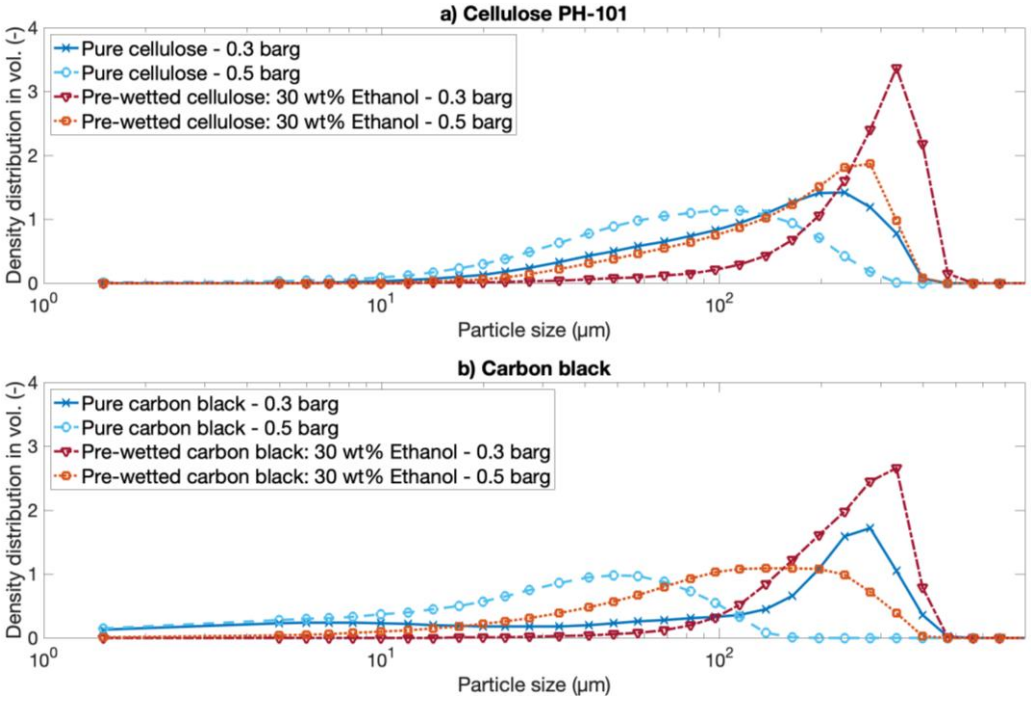

Figure 5: PSD evolution of pure dusts and powder/solvent admixtures at the exit of a Godbert-Greenwald oven as a function of the dispersion pressure

Once again, the behaviors of cellulose and carbon black mixed with ethanol are strongly different: the MIT of cellulose decreases slightly from 440 to 430 °C when 25 wt.% solvent is added, whereas the MIT of carbon black drops from 690 to 450 °C with the same addition. A similar trend is observed for soil/diesel mixtures. The decay is non-linear and tapers off at higher concentrations, which is probably related to powders agglomeration. However, it should be noted that the MIT changes as function of the dispersion pressure. A few tests were

performed with water, obviously leading to an increase in MIT. It is interesting to note that this increase is generally of the same magnitude as the decrease observed when solvent is added.
Figure 5a shows that the PSD of pure cellulose slightly increases when injected in a Godbert-Greenwald oven compared to its initial PSD shown in Table 1, which was already highlighted by Pietraccini et al., 2024. It also demonstrates that the pre-wetting of the cellulose strongly increases its PSD, especially at low dispersion pressures. Pre-wetted cellulose containing 30 wt.% ethanol and dispersed at 0.5 barg has a PSD comparable to that of pure cellulose dispersed at 0.5 barg, which confirms that the minimum ignition temperature can be found at different dispersion pressures as a function of the solvent content.
A similar PSD change is observable for carbon black. However, it is worth noting that carbon black is initially a very fine powder prone to agglomeration, due to the presence of strong Van der Waals interactions between particles. Such phenomena can explain the significant PSD difference observed between 0.3 and 0.5 barg for pure carbon black. Even pre-wetted with 30 wt.% ethanol, the presence of fine particles is still noticeable at high dispersion pressures. Thus, pre-wetting a powder with a flammable solvent changes its PSD, which impacts both its residence time in the Godbert-Greenwald oven and its heating characteristic time.

4. Conclusions

Both explosion severity and minimum ignition temperature can be significantly modified when pre-wetting a powder. In terms of risk assessment, it is not possible to consider this type of admixture to be equivalent to a hybrid gas/solid mixture of the same composition. Changes in PSD, in dust cloud turbulence, as well as the effect of solvent vaporisation on powder heating kinetics, need to be considered. However, it appears that the increase in PSD due to granulation generally has a negative impact on the explosion severity of the admixture at high solvent concentrations. The promotional effects of flammable solvents depend greatly on the nature of the powder: its shape (fiber for cellulose, sphere for carbon black), its solvent affinity (e.g. swelling of cellulose in ethanol), its size, its porosity, its calorific capacity (low for carbon black) or its chemical composition (e.g. presence of oxygen in cellulose unlike carbon).
The state of the mixture and the concentration of solvent being variable throughout the process, this study improves the identification of zones at higher explosion risk.

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