

IGNITABILITY AND EXPLOSIBILITY OF POLYESTER/EPOXY RESINS FOR ELECTROSTATIC POWDER COATING

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Summary

The use of electrostatic powder coating is expanding. In view of the dust explosion hazard related to this process, a comprehensive investigation of ignitability and explosibility properties of 11 polyester/epoxy resin powders used in electrostatic powder coating has been carried out.

The powders differed with respect to the ratio of polyester to epoxy, pigment type, pigment content, density and particle size distribution.

The powders were tested in the closed Hartmann bomb for establishing the maximum explosion pressure and the maximum rate of pressure rise, in the open Hartmann tube fitted with the CMI electric spark generator for measurement of the minimum ignition energy, and in the 'Nordtest Fire 011' apparatus for determination of the minimum explosible dust concentration. Attempts were also made at conducting some explosibility tests in the Swiss closed 20-litre spherical vessel, but severe blocking problems in the dust dispersion system were encountered. Particle size distributions of the powders were determined using a laser diffraction method, and the specific surface areas were measured by nitrogen adsorption.

All the powders gave approximately the same maximum explosion pressures in the Hartmann bomb, whereas the maximum rate of pressure rise decreased with increasing pigment content and particle size. Clouds in air of all the resins had quite low minimum ignition energies, from below 3 mJ to approximately 20 mJ. Except for one or two powders the minimum ignition energy increased fairly systematically with particle size. There was no systematic influence of the pigment content, although the powders with the highest pigment contents also had the highest minimum ignition energies. The minimum explosible dust concentration increased systematically with increasing pigment content, in such a way that the concentration of combustible material at the minimum explosible dust concentration was nearly the same for all the dusts, and close to the minimum explosible concentration of gaseous hydrocarbons like methane and propane.

2. Background

The technique of electrostatic powder coating is almost 20 years old, and its share of the market is growing. Powder coatings in common use are epoxy, polyester, polyurethanes and combinations of these. Application is by electrostatic spraying.

From a fire hazard point of view, there is a distinct difference between solvent-based paints and powder paints. While powder paints contain no combustible liquid solvents, the powder itself can represent an explosion hazard.

Although this has not been confirmed experimentally, it cannot be excluded that powder paint explosions may be initiated by mechanical sparks caused by the careless handling of equipment and tools. Glowing cigarettes could be another source. Objects that are not earthed can act as capacitors giving off dangerous electric discharges. Energy for ignition could also be provided by the electrostatic discharge between an earthed object and a spray gun. Manufacturers of spray guns have attempted to counteract this by equipping guns with a protective device that reduces the amount of current if the electric field between the gun and the object becomes too strong. When the gun comes too close, the current is cut off, but will be automatically switched on again as soon as the gun is removed sufficiently far away from the object. When properly adjusted, this system should provide efficient protection against spark discharge between the gun and the object.

Because complete elimination of potential ignition sources is difficult to achieve, safety regulations for electrostatic powder coating systems include strict control of the ventilation in the powder coating chambers. In Sweden the air flow in chambers shall be matched to the flow of powder paint so that the average concentration of powder suspended in the air in the chamber at any time does not exceed half the minimum explosible dust concentration. If the minimum explosible dust concentration is not known, the amount of powder shall not exceed 5 g/m^3 air [1].

West German regulations limit the powder/air mixture to a maximum of 10 g/m^3 when the minimum explosible dust concentration is unknown [2]. According to the NFPA Fire Codes, the corresponding value in the USA is 15 g/m^3 [3].

The minimum explosible dust concentration values used in these assessments are normally taken from published tables containing figures that have been determined for pure polymers with very fine size particles. This means extremely low minimum explosible concentrations. Published values for the maximum rate of pressure rise and the maximum pressure are also frequently extreme values for pure and very fine polymers.

There have been some indications that powder paints in current use have less severe dust explosion properties than those appearing in published tables. Although one would expect that both addition of inorganic pigments in the polymer and increasing the particle size will influence the dust explosion characteristics, it has not been possible to trace any published work aimed at quantifying these influences systematically.

If the maximum permissible dust concentration in powder coating chambers could be increased, improvements should be experienced in the actual coating process. The somewhat slower recirculation of air would provide a less turbu-

lent air environment in the chamber and thereby improve the ability of the powder to reach in accessible corners of the object to be coated. Efficiency would be improved by reducing overspraying. This would in turn reduce the fire and explosion hazard because of the smaller quantity of powder being recirculated. This is an important aspect, because the recirculated powder will have a finer size distribution than the main bulk of powder and therefore be more hazardous.

One main objective of the present work has been to investigate whether easing the ventilation requirements can be justified for some powder categories.

3. Experimental methods used

The dust samples were tested in the closed Hartmann bomb for establishing the maximum explosion pressure and the maximum rate of pressure rise [4]. An attempt was also made at testing some of the powders in the 20-litre spherical explosion bomb designed by Bartknecht and Siwek [5] for establishing an alternative set of maximum explosion pressures and maximum rates of pressure rise that would comply with the new ISO standard [6]. However, this was unsuccessful because of severe problems with the dust dispersion. A substantial fraction of the powder to be dispersed in the vessel remained inside the dispersion ring. As a result the average dust concentration of the cloud in the vessel was not known. Besides, the blocking of the ring necessitated time consuming dismantling of the dispersion system and careful cleaning of the horn between each individual test. This kind of problems with the Swiss 20-l vessel, which have been reported previously by other workers [7], are likely to become particularly severe when testing the type of electrically insulating powders studied in the present investigation.

The question of how the dust explosion violence should preferably be determined remains to be answered. A comprehensive discussion of the problem was given recently by Eckhoff [8]. Presently there exists no laboratory test method that is capable of predicting the explosion violence in all practical situations in industry. Data from any existing closed-bomb test must therefore be used with care.

The open Hartmann apparatus, fitted with the CMI electric spark generator [9], was used for establishing the minimum ignition energy of a dust cloud. This method is in agreement with the method recommended recently by a group of large chemical companies in Europe, TNO in the Netherlands, and CMI [10].

The minimum explosible dust concentration was determined using a simplified version of the 'Nordtest Fire 011' method [11]. The simplification was that the local dust concentration in the vicinity of the ignition source was not measured directly. Instead it was assumed that the dispersed dust was, on the whole, distributed evenly throughout the explosion space at the moment of ignition.

Local variations of concentration in the vicinity of the ignition source, would, however, be expected from test to test. In view of the fact that the transition of the probability of ignition from zero to hundred per cent normally occurs over a significant range of nominal dust concentrations, it was necessary to identify a certain probability by which the minimum explosible dust concentration should be defined. In the present case, 50% probability was chosen on the basis of the following assumptions. The first was that the lack of reproducibility of the local dust concentration in the region of the ignition source at the moment of ignition, in a series of nominally identical experiments, was the dominating uncertainty factor. Secondly, it was assumed that the variation of this local concentration around the arithmetic mean value in a series of experiments was symmetrical (e.g. Gaussian). Finally, since the local dust concentration was not measured directly, it had to be assumed that at the moment of ignition, the average local dust concentration was approximately equal to the overall nominal dust concentration (dispersed dust mass divided by the vessel volume). It then follows that the nominal dust concentration in a series of replicate experiments that gave 50% probability of ignition was the best estimate of the true minimum explosible concentration. It should be briefly mentioned that Nordtest Fire 011 [11] does specify a method for direct measurement of local dust concentration. Experience has shown, however, that this method is not satisfactory, and an improved method is currently being considered for inclusion in the test [12].

The particle size distributions of the dust samples were determined by means of the Sympatec Helos laser diffraction pattern analyser [13]. Measurements of the specific surface of the powders were carried out using the Ströhlein nitrogen adsorption technique [14].

4. Properties of the powders investigated

All the powders investigated were polyester/epoxy resins. The various data for polyester/epoxy content, pigment content, pigment type, and particle density are given in Table 1.

5. Results

5.1 Maximum explosion pressure and rate of pressure rise

The results of the Hartmann bomb tests are given in Table 2, which also gives the dust concentrations at which the highest values of the maximum pressure and maximum rate of pressure rise were obtained.

5.2 Minimum ignition energy of a dust cloud

The results are given in Table 3.

TABLE 1

Some physical and chemical data for the polyester/epoxy powders investigated

No.	Powder	Polyester/ epoxy (%)	Pigment type	Pigment volume concentration (%)	Density (g/cm ³)	Non- combustible mass fraction (%)
1	White A	75/25	titanium oxide	21.1	1.70	42.61
2	Blue B	0/100	cobalt oxide chromium oxide aluminium oxide	24.3	1.70	47.12
3	Yellow C	30/70	iron oxide	9.3	1.39	21.12
4	Red D	97/3	iron oxide	4.4	1.35	11.40
5	Grey E	50/50	iron oxide	15.15	1.65	35.24
6	Clear F	100/0	no pigment	0	1.16	0
7	Brown G	75/25	iron oxide	20.11	2.00	50.62
8	Blue H	75/25	cobalt oxide chromium oxide aluminium oxide	20.11	1.85	46.70
9	Red I	75/25	organic	30.07	1.40	0
10	White J	0/100	calcium carbonate titanium oxide	14.05	1.59	36.02
11	White K	0/100	calcium carbonate titanium oxide	14.05	1.59	36.02

TABLE 2

The results from the Hartmann bomb tests

No.	Powder	P_{\max} (bar(g))	Concentration (g/m ³)	$(dP/dt)_{\max}$ (bar/s)	Concentration (g/m ³)
1	White A	6.3	1000	190	750
2	Blue B	5.4	800	150	750
3	Yellow C	6.0	1000	240	500
4	Red D	5.4	1500	270	750
5	Grey E	5.9	1200	180	450/1100
6	Clear F	5.6	750/1500	180	750/1500
7	Brown G	5.9	1400	118	750
8	Blue H	6.0	2000	115	750
9	Red I	7.2	1300	175	500/1000
10	White J	6.4	750	200	750
11	White K	6.4	700	410	750

TABLE 3

Minimum electric spark ignition energies of clouds in air of various polyester/epoxy powders

No.	Powder	MIE (mJ)
1	White A	<3
2	Blue B	10-15
3	Yellow C	<3
4	Red D	3-7
5	Grey E	<3
6	Clear F	<3
7	Brown G	15-25
8	Blue H	20-25
9	Red I	6-9
10	White J	15-22
11	White K	<3

TABLE 4

Minimum explosible dust concentrations of various polyester/epoxy powders

No.	Powder	MEC (gm ⁻³)
1	White A	58
2	Blue B	60
3	Yellow C	40
4	Red D	42
5	Grey E	49
6	Clear F	35
7	Brown G	63
8	Blue H	67
9	Red I	33
10	White J	45
11	White K	40

5.3 Minimum explosible dust concentration

The results are given in Table 4.

5.4 Particle size distribution

The particle size distributions determined by the Sympatec Helos apparatus are given in Table 5.

5.5 Specific surface area

Specific surface areas of the powders were determined both by the Ströhlein nitrogen adsorption method and by calculation from the particle size distributions assuming spherical particles.

TABLE 5

Particle size data for various polyester/epoxy powders

No.	Powder	Particle diam. (μm) below which 10, 50 and 90% of the total mass of particle falls		
1	White A	11	33	60
2	Blue B	17	69	138
3	Yellow C	9.6	32	69
4	Red D	15	48	104
5	Grey E	12	40	82
6	Clear F	8.7	35	72
7	Brown G	15	42	84
8	Blue H	20	59	123
9	Red I	17	54	119
10	White J	22	59	114
11	White K	8	23	49

TABLE 6

Specific surface areas of various polyester/epoxy powders determined by nitrogen adsorption and by calculation from the particle size distributions assuming spherical particles

No.	Powder	Specific surface area from nitrogen adsorption (m^2/g)	Specific surface area calculated from particle size distribution (m^2/g)	Ratio
1	White A	0.47	0.16	2.9
2	Blue B	0.74	0.10	7.4
3	Yellow C	1.59	0.22	7.2
4	Red D	0.25	0.15	1.7
5	Grey E	0.32	0.15	2.1
6	Clear F	0.34	0.36	0.94
7	Brown G	0.26	0.11	2.4
8	Blue H	0.30	0.09	3.4
9	Red I	0.41	0.13	3.2
10	White J	0.30	0.10	3.0
11	White K	0.48	0.23	2.1

For a given powder the ratio between the specific surface area from the Ströhlein measurement and that calculated from the particle size distribution, gives information about the shape of the particles in a powder (assuming non-porous material, and complete dispersion of the particles in the size analysis tests).

The two sets of specific surfaces are given in Table 6. With the exception of

powder 6, the Ströhlein area was always larger than the equivalent sphere areas, as would be expected. For powder 6 the particles were probably close to spherical and well dispersed in the Helos test. The other results seem to fall in two main groups. Powders No. 1, 4, 5, 7, 8, 9, 10 and 11 have area ratios mostly in the range 2–3, whereas powders 2 and 3 have comparatively high ratios of more than 7. The available data do not permit any further discussion of possible reasons for these differences. Scanning electron microscopy would probably provide valuable additional information.

6. Dependence of explosibility and ignitability on particle size and pigment content

6.1 Maximum explosion pressure

The results from the Hartmann bomb tests (Table 2) show, with the exception of powder No. 9, that the maximum explosion pressures are within the range 5.4 to 6.4 bar (g) for all the powders. One reason for powder No. 9 giving a somewhat higher pressure than the others, could be that organic pigment was used in this powder, whereas the pigments in all the other pigment containing powders were inorganic. The inorganic pigments will act as heat sinks, and thus contribute to the lowering of the explosion pressure, whereas the organic pigment burns and contributes to the production of heat. However, powder No. 6, containing no pigment at all, did not give any higher pressure than the powders containing inorganic pigments. Therefore, the cause of the higher maximum explosion pressure of powder No. 9 remains to be identified.

6.2 Maximum rate of pressure rise

Influence of non-combustible material

Maximum rate of pressure rise versus non-combustible mass fraction is plotted in Fig. 1.

Figure 1 indicates a slight reduction of the maximum rates of pressure rise with increasing content of non-combustible material from 10% and upwards. However, both of the powders containing combustible material only have comparatively low maximum rates of pressure rise. The reason for this is not clear. The comparatively high rate of pressure rise of powder No. 11 can, as will be shown below, be explained in terms of particle size.

Influence of particle size

The maximum rate of pressure rise for powders of any given material normally increases systematically with decreasing particle size. This is because a smaller particle size yields a larger interface area between solid material and air per unit mass of powder, and therefore the combustion rate will be higher. Figure 2 shows the maximum rate of pressure rise versus the median particle

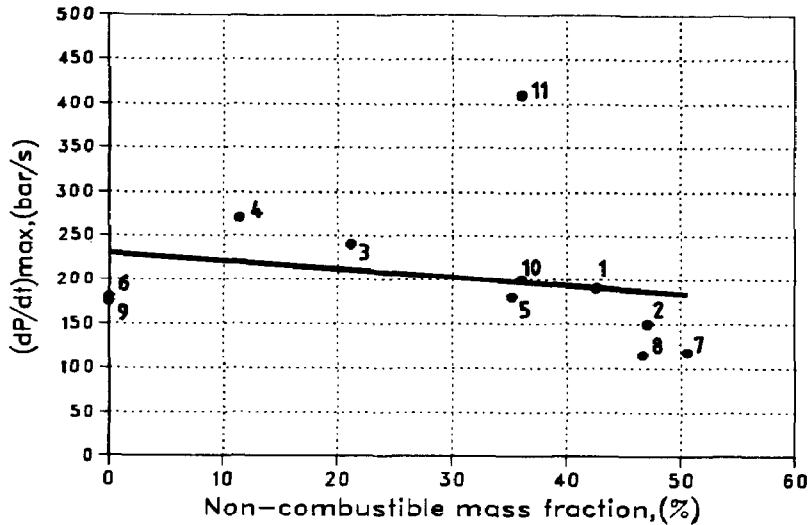


Fig. 1. Maximum rate of pressure rise versus non-combustible mass fraction.

diameter for the polyester/epoxy powders. The specific effect of particle size is demonstrated by powders No. 10 and No. 11, which are chemically identical the only difference being the particle size. The coarser powder, with a median particle diameter of $59 \mu\text{m}$, has a maximum rate of pressure rise of 200 bar/s, whereas the value for the finer powder, with a median particle diameter of $23 \mu\text{m}$, is 410 bar/s.

The scatter of the other data points in Fig. 2 reflects the influence of chemical composition and possibly also particle shape.

6.3 Minimum ignition energy of clouds in air

Influence of non-combustible material

The relationship between the content of non-combustible material of the powders and the minimum ignition energy (MIE) is shown in Fig. 3. Due to basic limitations in the electric spark generator it was not possible to differentiate between the five powders having MIE-values below 3 mJ. For the other powders it seems that those having the highest minimum ignition energies were generally found among the ones having the highest contents of non-combustible material. However, some powders with as much as 35–45% non-combustible material also ignited by electric sparks of energies below 3 mJ. Figure 3 therefore indicates that the mass fraction of non-combustibles is not the primary parameter. The differences in the MIE-values can to a large extent be explained in terms of particle size.

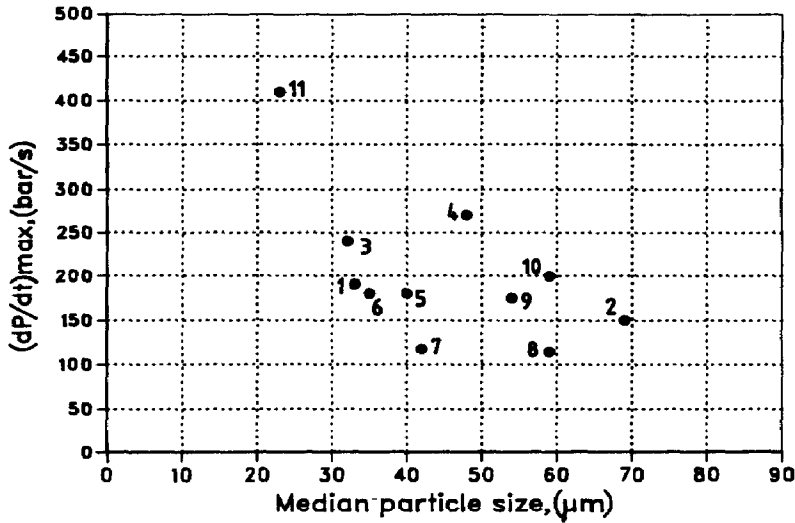


Fig. 2. Maximum rate of pressure rise versus median particle diameter, d_{50} .

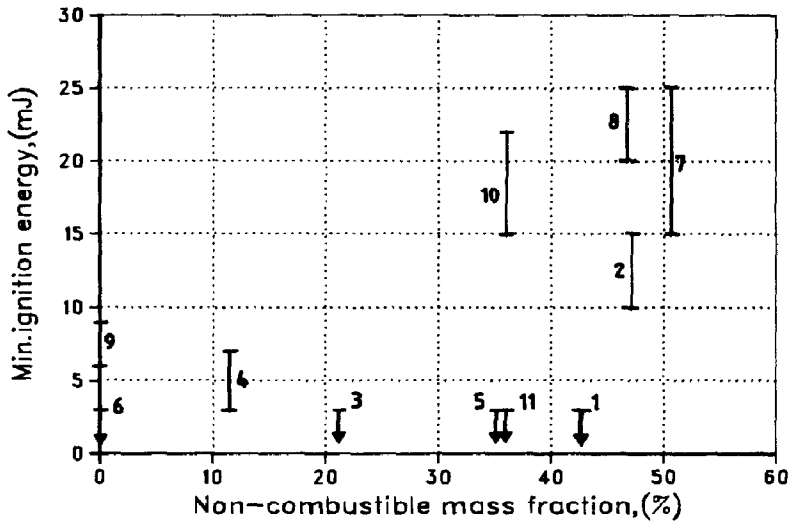


Fig. 3. Minimum ignition energy versus non-combustible mass fraction.

Influence of particle size distribution

The influence of particle size on minimum ignition energy is shown in Fig. 4. There is a clear tendency of the minimum ignition energy increasing with particle size in agreement with what has been found for other types of combustible powders and dust. All the powders of median particle size below $40 \mu\text{m}$ had minimum ignition energies below 3 mJ. As the median particle size in-

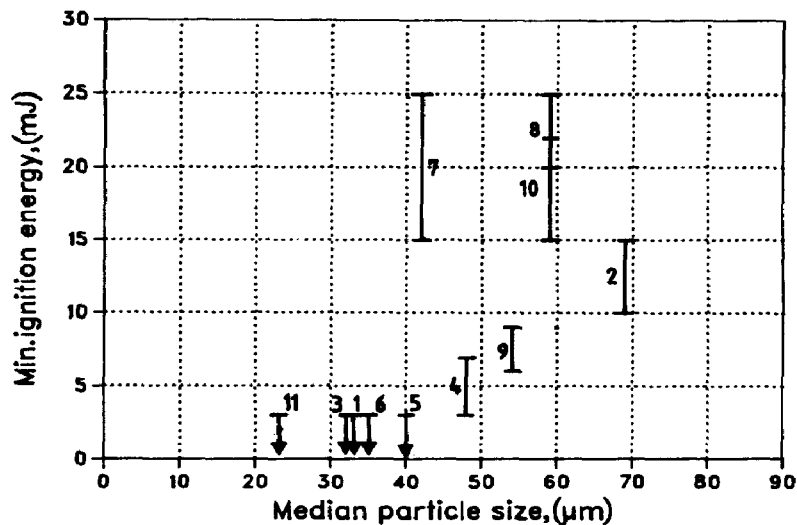


Fig. 4. Minimum ignition energy versus median particle diameter.

creased from 40 μm to 70 μm , MIE increased reasonably monotonically, except for powder No. 7. High pigment mass fraction might explain why this powder is out of line with the others. However, powder No. 2 contains almost the same fraction of pigment as No. 7, but has a lower MIE in spite of larger median particle size. A satisfactory explanation of the comparatively high MIE of powder No. 7 thus remains to be found.

6.4 Minimum explosible concentration

Influence of non-combustible material

The minimum explosible dust concentration versus content of non-combustible material is given in Fig. 5. There is a clear tendency that the minimum explosible dust concentration increases with increasing content of non-combustible material, i.e. increasing pigment content. Because powder No. 9 contains a combustible pigment, the content of non-combustible material is zero. If one assumes that the non-combustible material does not have any influence on the minimum explosible concentration of combustible material in the dust, the latter quantity should be about 32 g/m^3 irrespective of the non-combustible pigment content! If then p is the mass fraction of non-combustibles in percent, the gross minimum explosible dust concentration will be;

$$\text{MEC} = 32(100/(100-p)) \quad (\text{g}/\text{m}^3)$$

This relationship gives the curved line in Fig. 5, and it is seen that the agreement with the experimental points is reasonable. Approximate estimates of MEC for various contents of non-combustible material can be obtained by this relationship. However, undue extrapolation beyond the experimental points will give erratic results, as flame propagation through dust clouds will become impossible at p -values significantly lower than 100%.

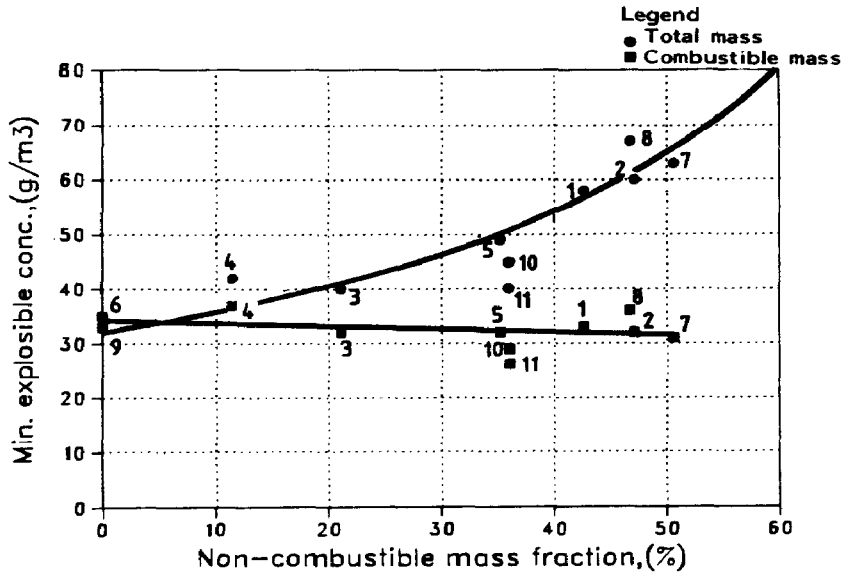


Fig. 5. Minimum explosible dust concentration versus non-combustible mass fraction.

Table 7 gives the mass fractions of non-combustible material in the various polyester/epoxy powders studied. These figures were used to transform the various minimum explosible concentrations measured to minimum explosible concentrations of combustible material as shown in Table 7. These values have also been plotted in Fig. 5.

The straight line through the square points is approximately horizontal, indicating that the minimum explosible concentration of combustible material is in fact almost constant and independent of pigment content. The chemical composition of the combustible substance does not seem to influence its minimum explosible concentration. In view of this it is of interest to compare the value of 31–35 g/m³ with published explosibility limits of gaseous hydrocarbon in-air mixtures. For methane-in-air and propane-in-air, the limits are approximately 5 vol.% and 2 vol.%, respectively. Converted to mass concentrations, this equals 33 g/m³ and 36 g/m³ respectively (at 25°C), which is quite close to the measured minimum concentrations of combustible material for the polyester/epoxy powders. This may indicate that the flame propagation through the dust clouds at the limiting concentration is similar to that through a pre-mixed gas, i.e. flame propagation takes place in combustible gas evolved from the particles in the preheating zone just ahead of the flame. This has previously been shown to be the case for flame propagation through fine liquid aerosols [15].

Evidence from determination of minimum explosible dust concentrations of epoxy powders in a large-scale vertical tube apparatus [16] indicates that the

TABLE 7

MEC of combustible material in dust clouds based on pigment mass fraction and the measured minimum explosible concentration

No.	Powder	Mass fraction of non-combustible material (%)	Measured min. expl. dust concentration (g/m ³)	Min. expl. concentration of combustible material (g/m ³)
1	White A	42.61	58	33.3
2	Blue B	47.12	60	31.7
3	Yellow C	21.12	40	31.6
4	Red D	11.40	42	37.2
5	Grey E	35.24	49	31.7
6	Clear F	0	35	35.0
7	Brown G	50.62	63	31.1
8	Blue H	46.70	67	35.7
9	Red I	—	33	33.0
10	White J	36.02	45	28.8
11	White K	36.02	40	28.6

figures in Table 7 are not grossly in error. The ignition source was a propane flame. The particular experiments with epoxy powders were communicated to the authors separately [17]. For a pigment-free powder, flame propagation occurred above 20 g/m³ and for a 'highly' pigmented powder above 40 g/m³. However, these figures most probably refer to the limits for very weak flames, propagating through only a part of the tube length. Propagation over the entire tube length of 5 m in this particular apparatus normally required concentrations of about twice the limits for marginal propagation, which would be about 40 g/m³ for the pigment-free powder and 80 g/m³ for that with 'high' pigment content. If it is assumed that realistic values for the practice would lie somewhere between these two extremes, such values would be very close to those in Table 7.

If the maximum permissible average concentration of powder in suspension in powder coating chambers is to be half the minimum explosible concentration, Table 7 shows that those limits will be from 17 g/m³ to 33 g/m³, depending on the mass fraction of combustible material. This is significantly higher than the maximum values currently permitted in Sweden [1] and West Germany [2], and in most cases also higher than the maximum limit in USA [3].

Influence of particle size distribution

The measured relationship between the minimum explosible dust concentration and the mean particle diameter is shown in Fig. 6. There is an indication that the minimum explosible dust concentration increases slightly with increasing particle size. However, the interpretation of this correlation is not

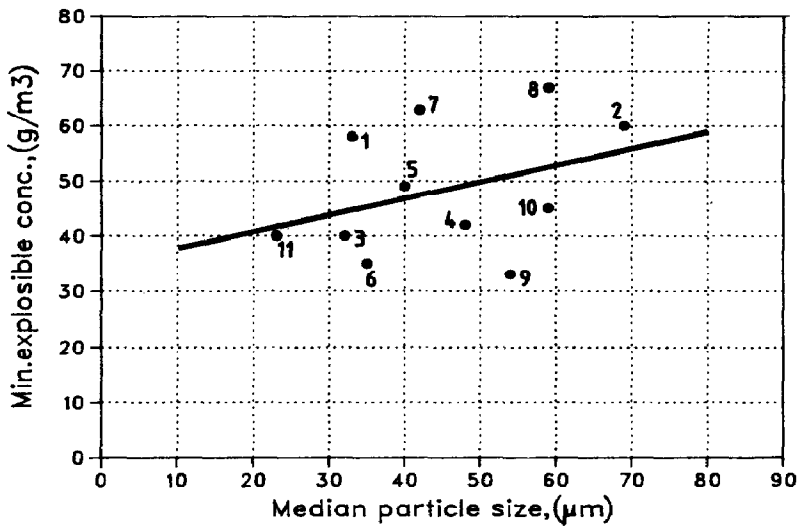


Fig. 6. Minimum explosible dust concentration versus median particle diameter, d_{50} .

straight-forward, because the correlation also reflects a variation of the content of non-combustible material with particle size. The fact that powders 10 and 11, which are chemically identical, have quite similar minimum explosible dust concentrations, suggests that the influence of particle size within the range considered is rather weak.

7. Conclusions

1. The maximum explosion pressures are approximately the same for all the powders, with the exception of one, irrespective of particle size and content of non-combustible material. This is in accordance with expectations in view of the similar heats of combustion per mole oxygen consumed of the organic materials of which the powders are composed.
2. For powders of the same chemical composition, the maximum rate of pressure rise increases systematically with decreasing median particle size as for other powder types. The influence of the content of non-combustible pigment on the rate of combustion is less clear.
3. The powders, when dispersed as clouds in air, have minimum ignition energies ranging from below 3 mJ to approximately 20 mJ. On the whole, the minimum ignition energy increases systematically with increasing particle size, irrespective of the variation of the content of non-combustible material.
4. The minimum explosible dust concentration increases systematically with increasing content of non-combustible material. The lowest minimum explosible concentrations of 33–35 g/m^3 were obtained for the powders containing combustible material only. The same value was also obtained for all

the other powders, if the minimum explosible concentrations were converted to minimum explosible concentration of combustible material only (obtained by subtracting the concentration of non-combustible materials from the measured minimum explosible concentrations). This common value is close to the minimum explosible concentrations for methane and propane in air.

5. In view of the very low minimum ignition energies of some of the powders and the significant explosibility in terms of maximum explosion pressure and maximum rate of pressure rise, one may wish to maintain the existing, fairly strict requirements to ventilation of coating chambers in terms of the maximum permissible quantities of fine powder fractions in recirculation. However, if one decides to maintain half the minimum explosible concentration as the maximum permissible limit, the present investigation indicates that these limits should generally be significantly higher than the values currently specified in Europe and USA.

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