

Analysis of susceptibility to ignition of dust layer and dust cloud of selected hardened unsaturated polyester resins

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Abstract: In the article there were described combustible properties of selected dust types formed of plastic materials. Those properties decide on fire and explosion hazard for dust in layer and in cloud, according to PN-EN 50281-2-1:2002. The article also consists of safety requirements concerning using appliances in explosion hazard areas, according to PN-EN 50281-1-2:2002. In the article there are presented results of investigations on minimal ignition temperature of layer (MITL) and minimal ignition temperature of dust cloud (MITDC) as a function of dust layer thickness of chosen industry dust with different addition of modification and values of maximum acceptable surface temperature (MAST) of machines operating in the presence of dust cloud and chosen dust layer with thickness of 5 and 12.5 mm.

Keywords: hardened unsaturated polyester resin, susceptibility to ignition, industry dust, dust explosion, industry safety.

Analiza podatności na zapalenie warstwy osiadłej i obłoku pyłów wybranych termoutwardzalnych nienasyconych żywic poliestrowych

Streszczenie: Oceniano właściwości palne wybranych pyłów przemysłowych powstały na bazie żywicy poliestrowej Polimal – produktu Zakładów Chemicznych Organika-Sarzyna w Nowej Sarzynie – decydujące o zagrożeniu pożarowo-wybuchowym pyłów w warstwie i w obłoku. W badaniach wykorzystano próbki o rozmiarze ziarna poniżej 200 µm, niemodyfikowanej utwardzonej żywicy poliestrowej Polimal 1033APy, żywicy Polimal 1033APy z dodatkiem 14 % mas. Sb₂O₃, żywicy Polimal 1033APy z dodatkiem 15 % mas. MoO₃ oraz pyłu akrylowego. Opisano warunki bezpiecznego użytkowania urządzeń stosowanych w strefach zagrożenia wybuchem, zgodnie z PN-EN 50281-1-2:2002. Wyznaczono wartość minimalnej temperatury zapłonu pyłu w warstwie (MTZW) i w obłoku (MZTO), w funkcji grubości warstw wybranych pyłów polimerowych (tabela 3) oraz maksymalne dopuszczalne wartości temperatury powierzchni (MDTP) urządzeń pracujących w obecności pyłów w warstwie o grubości 5 mm (tabela 4) lub 12,5 mm (tabela 5) oraz w obłoku (tabela 6).

Słowa kluczowe: termoutwardzalne nienasycone żywice poliestrowe, podatność na zapalenie, pyły przemysłowe, wybuchi pyłów, bezpieczeństwo przemysłowe.

Due to their properties plastic materials have diverse applications in almost every field of life. Despite their numerous advantages, a great disadvantage of plastics both in a solid and ground form is the fire and explosion hazard they cause. In almost all industrial plants, in which mechanical processes are being carried out, such as cutting, grinding, milling, slicing *etc.* of solids, dust becomes accumulated [1, 2]. Its presence gives rise to a hazard, as the appropriate ignition sources can cause a fire or explosion of dust, which may as a consequence lead to death or body harm [3–7]. Moreover, those phenomena can cause considerable property losses. In the event of a fire the most frequent cause of death or injury and loss of proper-

ty is the thermal radiation and toxic products of combustion and of thermal decomposition, and as regards an explosion, this comprises a pressure wave and fragments of materials. Over the recent years fires of plastic materials took place in Poland. Among other things there were fires in factory producing service packaging Polarcup in Siemianowice Śląskie in 2000, fire of polypropylene pile in a plant in Sieradz (2002), fire of a room in a polypropylene packaging recycling plant in Chrzązonowice (2008), a fire in Plastic Materials Processing Plant in Nowa Wieś near Kęty (2008), and fire in a storage of plastic pressed materials prepared to be recycled in Nysa (2008).

In Poland and other European countries legal regulations impose on the employer an obligation to verify whether dust that occurs in an enterprise poses an explosion hazard. If the possibility of such a hazard is ascertained, the employer has to undertake measures related to the evaluation of the explosion risk and apply all means

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to limit the risk to an admissible level (PN-EN 50281-1-2:2002, PN-EN 50281-2-1:2002). The occurrence of dust entails the accumulation of dust. Of particular importance is the prevention of accumulation of those layers on surfaces of machines and installations that become heated up, because the combustible dust that settles on them limits the exchange of heat between the devices and air, which as a consequence may cause its ignition. Consequently it is necessary to make sure that the temperature of particular machines does not come up to a level at which the settled dust could pose a fire and explosion hazard. The most hazardous types of dust with respect to fire and explosion hazards include first of all dust obtained from thermally hardened materials, among others from hardened polyester resins.

The execution of tests related to the determination of the minimum ignition temperature in a dust layer and in cloud allowed the definition of admissible temperature for devices operating at the presence of tested types of dust coming from plastic materials.

The objective of this paper is to carry out a comparative analysis of values of the minimum ignition temperature in a layer and in a cloud of selected dust types formed of plastic materials. It was based on tests executed in accordance with PN-EN 50281-2-1:2002.

EXPERIMENTAL PART

Materials

Four types of dust consisting of plastic materials were applied in experimental studies. The dust used in the tests had a diameter of 200 µm in accordance with the standard PN-EN 50281-2-1:2002. The three tested dust types were obtained from grinding of polyester materials in a laboratory vibrating mill. Those types of dust were obtained from hardened polyester resins produced by the Organika Sarzyna Chemical Plant in Nowa Sarzyna. Names of Polimal resins used to make the dust were as follows:

— Polimal 1033APy — hardened resin non-modified, neither physically nor chemically;

— Polimal 1033APy + 14 wt % of Sb₂O₃ — hardened resin with an admixture of flame retardant, *i.e.* antimony trioxide, at a concentration of 14 wt % as compared to the polymer mass;

— Polimal 1033APy + 14 wt % of MoO₃ — hardened resin with the admixture of flame retardant in the form of molybdenum trioxide, at a concentration of 14 wt % as compared to the polymer mass.

The Polimal 1033APy resin is frequently used in Poland, the most frequently in the construction industry, for example in manufacturing of floor levelling compounds, structural products *etc.* The letter symbol stands for the following: A — ecological resin with low styrene contents, Py — accelerated resin which does not require a cobalt accelerator.

Table 1 presents density, humidity, heat of combustion, beginning of thermal decomposition and ignition temperature of tested dust types. Experimental error is about ± 10 °C.

Methods of testing

In accordance with the standard PN-EN 50281-2-1:2002, two methods of measuring the minimum ignition temperatures of dust may be applied:

— Method A — this method comprises the determination of the minimum ignition temperature of a dust layer (MITL) with a specified thickness on a heating plate. Method A is particularly applicable to an analysis of a fire hazard in the case of usage of industrial machines having hot surfaces, which operate in the presence of combustible dust. The sample to be tested should be prepared to be homogenous and representative in relation to the dust material being tested. In compliance with the specified standard the tested dust sample has to be sieved on a metal sieve or a metal perforated plate with square openings having a nominal size of 200 µm. Should the necessity arises to test dust having a larger particle diameter, it is also admissible to have the dust materials sieved through a screen with a nominal mesh size up to 500 µm. Dust subjected to testing were in conformity to the standard PN-EN 50281-2-1:2002 and sieved through screens with holes having a nominal value of 200 µm.

Listing of percentage particle size distribution for analyzed polymeric dusts was presented in Table 2.

— Method B — this method comprises the determination of the minimum ignition temperature of the dust cloud (MITDC) or other ground materials. It is applied for industrial machines, during the operation of which

T a b l e 1. Listing of thermochemical parameters for the analyzed dusts

Type of tested dust	Density g/cm ³	Humidity %	Heat of combustion kJ/kg	Beginning of thermal decomposition (at heat rate 10 °C/min in the air), °C	Ignition temperature °C
Dust of hardened polyester resin 1033APy	0.60	0.076	26 636	180	308
Dust of hardened polyester resin + 14 wt % of MoO ₃	0.63	0.42	22 780	193	314
Dust of hardened polyester resin + 14 wt % of Sb ₂ O ₃	0.48	0.23	23 165	210	310
Dust of acrylic tubs	0.41	0.18	27 180	181	311

dust is generated in the form of a short-lasting cloud. This arises from the fact that the time of occurrence of dust inside the oven is short. It supplements method A.

T a b l e 2. Listing of percentage particle size distribution for analyzed polymeric dusts

Nominal size of openings mm	Dust of hardened polyester resin 1033APy	Dust of hardened polyester resin + 14 wt % of MoO ₃	Dust of hardened polyester resin + 14 wt % of Sb ₂ O ₃	Dust of acrylic tubs
Percentage particle size distribution, %				
1.000	100	100	100	100
0.200	42.2	23.0	23.9	42.8
0.100	36.3	6.1	9.6	21.8

Experimental works are performed with the use of standard test stands and apparatuses presented in Fig. 1.

Change of temperature in 5 mm dust layer of hardened polyester resin 1033APy on a hot plate and thermocouple position for the hot plate temperature presented on Fig. 2.

Recording the temperature of the dust layer starts at the moment when the ring is filled with dust. The testing is to be continued until the ignition. In accordance with the standard PN-EN 50281-2-1:2002, it shall be considered that the ignition of a dust layer has taken place when:

- 1) Glowing or flaming burning is observed, or
- 2) The measured temperature has achieved 450 °C, or
- 3) The measured temperature has exceeded the temperature of the heating plate, by 250 °C.

As regards points 1) and 2) ignition is considered not to have taken place if it is possible to prove that the reaction does not turn into a glowing or burning process. Glowing is an undeniable process which is the most frequent symptom of ignition of a dust layer.

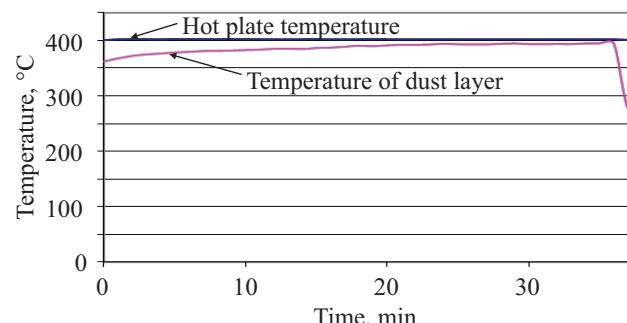


Fig. 2. Change of temperature in 5 mm dust layer of hardened polyester resin 1033APy on a hot plate (without ignition at 400 °C)

It is considered that ignition of a dust cloud has taken place if a flame is visible beyond the bottom end of the oven pipe. Nevertheless, a slight delay is admissible for ignition of a dust cloud. Sparks without a flame are not considered to indicate ignition of a dust cloud.

RESULTS AND DISCUSSION

The results of the executed tests related to values of minimum ignition temperatures for particular dust layers placed on a heating plate were presented in Table 3.

The obtained results indicate that the dust layer thickness is of importance both for the time until ignition, as well as for the minimum ignition temperature of dust. This may be proven by results obtained for dust made of hardened polyester resin 1033APy + 14 wt % of MoO₃. A 5 mm thick layer of this type of dust material was ignited at a temperature of 330 °C after a time of 26 min, while a layer 12.5 mm thick at a temperature of 310 °C after a time of 60 min.

As regards the 12.5 mm layer the dust temperature exceeded the plate temperature and amounted to 312 °C, while for a 5 mm thick layer ignition required reaching the temperature of 321 °C. Increasing the layer thickness



Fig. 1. Test stand for determination of minimum ignition temperature for: a) dust layer, b) dust cloud

T a b l e 3. Listing of collective values of the minimum ignition temperatures of a dust layer and the minimum ignition temperature of a dust cloud for the executed tests

Type of tested dust	MITL of a 5 mm layer °C	MITL of a 12.5 mm layer °C	MITDC °C	Time until ignition of a dust layer, min	
				5 mm	12 mm
Dust of hardened polyester resin 1033APy	>400	>400	500	none	none
Dust of hardened polyester resin + 14 wt % of MoO ₃	330	310	540	26	60
Dust of hardened polyester resin + 14 wt % of Sb ₂ O ₃	>400	400	520	none	55
Dust of acrylic tubs	>400	>400	520	none	none

Source: own study.

from 5 mm to 12.5 mm caused a reduction in the MITL for this dust material by 9 °C. As regards dust material of hardened polyester resin 1033APy + 14 wt % of Sb₂O₃, the layer thickness also affected the minimum ignition temperature. For the thinner layer (5 mm), layer ignition at the maximum oven temperature determined by the standard PN-EN 50281-2-1:2002 of 400 °C did not take place. Ignition of the layer took place only for 12.5 mm layer after a time of 55 min. For the two remaining types of dust material (of hardened polyester resin 1033APy and acrylic dust) ignition within the range of standard testing, *i.e.* up to an oven temperature of 400 °C for both layer thicknesses, has not taken place (Fig. 2).

Results of the conducted tests allow the presumption that the lowest value of ignition temperature for the tested dust materials was achieved for dust obtained from hardened Polimal 1033APy modified by molybdenum trioxide. As may be seen from available literature [8], molybdenum compounds in polymer materials tend to hasten the smouldering processes and increase the formation of carbonised layers on the surface of materials. The admixture of Sb₂O₃ neither affects smouldering nor leads to the lowering of the ignition temperature of dust material in relation to the dust obtained from non-modified material. Acrylic dust obtained in a tub technological process manifests similar properties concerning ignition of a dust layer from materials obtained from Polimal 1033APy, both non-modified and modified by Sb₂O₃.

The minimum ignition temperatures of a cloud of tested dust types differ slightly. For acrylic dust and dust obtained from hardened polyester resin Polimal 1033APy + 14 wt % of Sb₂O₃ the temperatures were equal and amounted to 520 °C. In addition, the optimum combustion conditions proved to be identical for all the tested types of dust material. Values respective for the mass of 1 g and pressure of 30 kPa proved to be the closest to stoichiometric concentration. Given the results obtained during tests of ignition of a cloud of certain dust materials, it may be presumed that the biggest susceptibility to ignition of a dust cloud was recorded for dust material obtained from a hardened non-modified resin, the next for dust obtained from a polyester material containing Sb₂O₃ and acrylic dust. The smallest susceptibility to ignition of a dust cloud as found for dust obtained from a polyester material with an admixture of MoO₃. The admixture of

antipyrenes applied to polyester materials reduced the explosion hazard of the analysed dust material obtained from hardened Polimal 1033APy. Molybdenum compounds, which have a significant impact on ignition of a layer of the analysed dust material (they lower the MITL), raise the MITDC as compared to the tested non-modified polyester material.

The conducted tests allow the determination of admissible temperatures on the basis of the standard PN-EN 50281-1-2:2002 for machines operating in the presence of analysed dust materials. The maximum temperature of the machine surface may not exceed a value lower by 75 °C than the MITL of the given dust material with a thickness of 5 mm, for a 12.5 mm layer this value is to be found on a diagram contained in the standard. Values of the MITDC are to be multiplied by 2/3 and the result of the index is the admissible value of the surface temperature of the given device. Tables 4–6 presented the admissible temperatures for machines for the tested dust materials on the basis of the MITL and the MITDC.

T a b l e 4. Admissible temperatures for devices operating at the presence of a 5 mm thick dust layer

Type of tested dust material	MITL of a 5 mm layer, °C	Admissible temperature for machines, °C
Dust of hardened polyester resin 1033APy	>400*)	>325*)
Dust of hardened polyester resin + 14 wt % of MoO ₃	330	255
Dust of hardened polyester resin + 14 wt % of Sb ₂ O ₃	>400*)	>325*)
Dust of acrylic tubs	>400*)	>325*)

*) As the Minimum Ignition Temperatures of a Dust Layer has not been determined, it is impossible to define the exact admissible temperature for machines.

Source: own study.

The maximum surface temperature of machines operating in the presence of a 5 mm dust layer may nevertheless be set out precisely on the basis of experimental testing for dust obtained from hardened polyester resin 1033APy + 14 wt % of MoO₃, and it is expected that it

T a b l e 5. Admissible temperatures for devices operating at the presence of a 12.5 mm thick dust layer

Type of tested dust material	MITL of a 12.5 mm layer °C	Admissible temperature for machines, °C
Dust of hardened polyester resin 1033APy	>400*)	>260*)
Dust of hardened polyester resin + 14 wt % of MoO ₃	310	140
Dust of hardened polyester resin + 14 wt % of Sb ₂ O ₃	400	260
Dust of acrylic tubs	>400*)	>260*)

*) As the Minimum Ignition Temperatures of a Dust Layer has not been determined, it is impossible to define the exact admissible temperature for machines.

Source: own study.

T a b l e 6. Admissible temperatures for machines operating in the presence of a dust cloud

Type of tested dust material	MITDC, °C	Admissible temperature for machines, °C
Dust of hardened polyester resin 1033APy	500	333.3
Dust of hardened polyester resin + 14 wt % of MoO ₃	540	360.0
Dust of hardened polyester resin + 14 wt % of Sb ₂ O ₃	520	346.7
Dust of acrylic tubs	520	346.7

Source: own study.

should be equal to 255 °C. For the remaining analysed dust materials the maximum surface temperature of machines operating in the presence of such dust materials in a layer 5 mm thick cannot be set out accurately as the MITL have not been determined. For a 12.5 mm thick dust layer the maximum temperatures of machines working in its presence may only be determined for dust obtained from hardened polyester resin 1033APy + 14 wt % of MoO₃ (140 °C) and for dust material obtained from hardened polyester resin 1033APy + 14 wt % of Sb₂O₃ (260 °C). Temperatures of machines that operate in the presence of dust clouds differ slightly. The lowest admissible surface temperature of a machine operating in the presence of a dust cloud is to be determined for dust material obtained from hardened polyester resin 1033APy, and it should be equal to 333.3 °C, while the highest one – for dust material obtained from hardened polyester resin 1033APy + 14 wt % of MoO₃ (360 °C). As regards dust material obtained from hardened polyester resin 1033APy + 14 wt % of Sb₂O₃ and acrylic dust, the admissible surface temperature of a machine operating in the presence of a cloud of such dust is the same and amounts to 346.7 °C.

The consequence of such high temperatures of working apparatuses are values of obtained experimental

results related to minimal temperatures in layer. Probably it is caused by the fact that sample with MoO₃ has faster ignition (as smouldering phenomena in undergoes thermal decomposition faster in base polymer) and flame retardant has greater tendency to stay in solid residues of thermal decomposition than in gas phase during combustion.

Conditions of the executed tests were in accordance with the standard and had an influence on the obtained minimum ignition temperatures of dust layers and clouds. The accuracy of the obtained results was affected by ovens used for the measurements. For measurements of the minimum ignition temperature of a layer this was of a much smaller importance than on the ignition temperature of a cloud.

CONCLUSIONS

— It turned out that the most hazardous dust with respect to fires was dust material obtained from hardened polyester resin 1033APy + 14 wt % of MoO₃, as both ignition temperatures of a 5 mm dust layer (330 °C) and of a 12.5 mm layer (310 °C) are the lowest.

— The most hazardous dust with respect to the cloud ignition proved to be dust material obtained from non-modified hardened polyester resin 1033APy (500 °C).

— The increase in the thickness of the dust layer reduces the ignition temperature of the dust layer, which may be proven by results obtained for dust material obtained from hardened polyester resin 1033APy + 14 wt % of MoO₃ and for dust material obtained from hardened polyester resin 1033APy + 14 wt % of Sb₂O₃. This arises from a more intense heat accumulation in the 12.5 mm dust layer as compared to a 5 mm layer.

— Ignition in a 12.5 mm dust layer takes place after a time longer than the one anticipated in a standard test (after 30 min the test is to be repeated at a higher temperature).

— Heating up of polyester dust of MoO₃ and Sb₂O₃ from hot surface of the analysed dust layers takes place at lower temperatures of the surface, but within a longer heating time of those layers.

— The addition of molybdenum compounds to polymer materials causes the acceleration of the smouldering processes, and also leads to lowering of the ignition temperature of the layer of tested dust material.

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