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Analysis of the Ability to Spontaneous Combustion and Ignition from Hot Plate of Dried Sewage Dust

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As of 2030 Directive (EU) 2018/850 introduces restrictions concerning landfilling of all waste suitable for recycling or other recovery of materials or energy, and in general, this legal act aims to limit the amount of municipal waste landfilled to 10% by 2035. The dangerous process of storing sewage sludge in the form of landfills requires the identification of flammable properties, among others of dried sewage dust, which has the capacity to ignite and to spontaneously combust during storage in silos. Hence, the aim of the study was to experimentally determine selected flammable properties of the sewage dust selected for testing, and to show the hazards that occur during their mass storage. The study determined the minimum ignition temperatures of layers and clouds of the tested sewage dust at constant temperature setting of the furnace surface in accordance with EN ISO/IEC 80079-20-2:2016 and at a constant increase in the furnace temperature of 3 °C/min in accordance with EN 60079-14:2014. A self-heating and self-ignition analysis of the deposited sludge dust was performed according to EN 15188: 2021 and a thermogravimetric test according to EN ISO 11358-1: 2014, and additionally the burning heat was determined according to EN ISO 1716:2018. In order to determine the possibility of reducing the susceptibility of dried sewage to self-heating, which leads to spontaneous combustion, two selected extinguishing powders for extinguishing class A, B, C and D fires were used in the tests. The minimum ignition temperature of the sewage sludge dust layer at constant temperature of the furnace surface with a layer thickness of 5 mm is 270 °C, and for a layer thickness of 12.5 mm – 250 °C. ABC Favorit was found to be the most effective extinguishing powder, and increased the value of the minimum ignition temperature of the layer (5 mm thick) to 360 °C. The self-ignition temperature value depends on the volume of the stored material. For the biggest studied volume (100 cm^3), the lowest self-ignition temperature was recorded which equalled to 144 °C. An analysis of the obtained test results allows the presumption that the dried sewage dust is a combustible material with properties similar to combustible materials of organic origin (biofuels).

1. Introduction

The intense development of sewer networks, construction of new sewage treatment plants and modernisation and extension of existing ones is expected to cause an increasing amount of municipal sewage sludge that requires management (Gao at all. 2014, Xu at all. 2013). Municipal sewage sludge is defined as sludge from digesters and other installations used for the treatment of municipal sewage and other sewage similar in composition to municipal sewage (Spinosa and Vesilind, 2001). Directive (EU) 2018/850 imposes landfill restrictions on all recyclable or otherwise recoverable materials or energy from 2030 onwards and generally seeks to reduce the amount of landfills storing municipal waste to 10% by 2035 (Directive 2018/850). According to Directive 86/278/EEC of the European Parliament and of the Council of 12 June 1986, sludge from which dried matter is produced is a mixture of water and solids separated from various types of waste water as a result of natural or artificial processes(Directive 86/278/EEC).The suspension of organic and inorganic solids in the waste water treatment process is subjected to treatment processes before it may be re-used. The dried sludge is stored in specific conditions to reduce any remaining pathogens and odours, and only after that process it can be considered for re-use (Aganetti at al., 2016, Nammari at all. 2004,). The chemical composition of sewage

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sludge depends on a number of factors, which include the scale of the agglomeration and its degree of industrialisation, and the proportion of industrial effluent in the treatment process (Abbas at all. 1994). However, for each individual process in which sewage sludge is treated, it is important to determine the combustion properties of dried sewage sludge, which has the potential to ignite and to self-ignite when stored in silos (Carras and Young, 1994, Higuera at all. 1989, Rynk, 2000). Given that it contains more than 50% organic substances, sewage sludge poses a fire hazard similar to that of biomass. During the drying or storage process, the sludge can become self-heated, which impairs its quality and a loss takes place of its mass (Babrauskaus, 2003). Continuous uncontrolled process of self-heating may lead to spontaneous combustion, which is highly dangerous for plants that produce dried sludge or those using it as an alternative fuel, as well as for the natural environment (Jafari at all. 2017). The occurrence of spontaneous combustion is influenced among others by such factors as grading, proportion of volatile parts, proportion of moisture, chemical composition and the temperature of the dried sludge during storage (WU, 2011, Xiaofang at all. 2018). Residual spores of microorganisms present in sewage granulate can develop in the "granulate-air-moisture" system and in certain conditions may cause the reactivation of biological activity. The resulting heat stored in the material catalyses the biological processes up to a certain temperature (self-heating). Above this temperature, chemical reactions take place, e.g. self-oxidation, leading to spontaneous combustion of the dried product. The process of self-heating and self-ignition occurs when there is a positive heat balance in the combustible system, i.e. when the amount of heat produced in the dried sewage sludge exceeds the amount of heat given off to the environment (Veznikova at all. 2014, Poffet at all. 2007). The causes of self-heating of dried sewage sludge and its subsequent ignition can also include low-temperature oxidation. Depending on the temperature range and the availability of oxygen, the following stages may be distinguished in this phenomenon: self-oxidation, smouldering, glowing and flame combustion . The absorption of water from the environment contributes to the temperature rise of the stored material and the energy gained is not quickly dissipated as the air between the dried particles is a good insulator (Półka, 2018, Eckhoff, 2019). Temperature rises also occur when the dried particles move between each other as a result of friction, and pressure may also rise when higher layers of dried product are pressed against lower layers, for example (during storage in a silo). The reason for the susceptibility of dry material to pressure changes may include the content of air and volatile parts (Zassa at all. 2013, Ptak and Półka, 2018). Therefore, the aim of this study was to experimentally determine selected flammability properties of sewage sludge and show the hazards that occur during its storage in a mass.

2. Experimental material

Dewatered sludge, a by-product of municipal and industrial wastewater treatment, was used to carry out the study. The content of organic matter in sewage sludge varies and depends on the water content of the sludge. Being a heterogeneous material, it consisted mainly of organic substances, biogenic elements e.g. carbon, hydrogen, nitrogen, oxygen, phosphorus sulphur as well as heavy metals and microorganisms. The dewatered sludge mass contained more than 52% organic matter. In order to determine the possibility of reducing the susceptibility of dried sewage sludge to the process of igniting the dust layer, two selected fire extinguishing powders designed to extinguish fires of classes A,B,C and D were used in the tests at a concentration of 20% by weight of the sludge dust. Physical properties of the tested sewage dry dust and basic technical data of the extinguishing powders used for the tests are presented in Table 1.

Table 1: Physical properties of the tested sewage dry dust and basic technical data of the extinguishing powders used

Substance name	Grinding, μm	Dust moisture, %	Bulk density, g/cm^3	Active substance in extinguishing agent/content, % weigh	Main type of extinguishing activity
Dust of dried sewage sludge	≤200	4.98	0.72	---	---
ABC Favorit extinguishing agent	≤200 μm	2.01	0.93 ± 0.07	monoammonium phosphate; ammonium sulfate / 21.50 ± 1.5; 74.0 ± 3.0	heterophasic inhibition, formation of an insulating layer (type A fires)
ABC Favorit extinguishing agent	≤200 μm	2.03	1.04 ± 0.04	alkali chloride / 90 ± 3.0	formation of a tight shell on the surface of a combustible material

The trade names of the extinguishing agents are ABC Favorit and D Favorit - Protekta product (Safety data sheet ABC Favorit, 2012, Safety data sheet D Favorit, 2012). The ABC Favorit extinguishing agent is dedicated for extinguishing the fires of types A, B and C, while D Favorit - for extinguishing the type D fire.

3. Test methods used to determine the combustibility properties of dried sewage sludge

In the study a determination was made of the minimal ignition temperature of layer (MITL) and of clouds of the tested dust (MTCD) of sludge and at constant setup of furnace surface temperature according to EN ISO/IEC 80079-20-2:2016 and at a constant furnace temperature rise of 3 °C/min according to EN 60079-14:2014 (EN ISO/IEC 80079-20-2, EN 60079-14). Under EN ISO/IEC 80079-20-2, the basic dust layer is to be formed with the use of a metal ring placed on the hotplate 5 mm high and the tests should be commenced by heating the hotplate to the chosen temperature for the measurement at constant temperature, while the initial setting of the hotplate needs to be 50-60°C for the measurement at constant temperature rise (PN-EN 60079-14:2014). The test is to be carried out until the dust layer is ignited in line with PN-EN ISO/IEC 80079-20-2-2016. The determination of MITL at a constant increase of the hotplate temperature was continued until the dust layer ignited in accordance with the criteria contained in PN-EN ISO/IEC 80079-20-2-2016-05. Tests for the establishment of the spontaneous ignition temperature in dust stored in volume/mass were executed pursuant to EN 15188:2021 (EN 15188). Dust spontaneous combustion occurs when the temperature at the geometric centre of the test has increased by at least 60 K above the hot storage temperature, when the time variation of the temperature at the geometric centre of the sample being tested indicates an inflection point, and this occurs at a temperature above the ambient temperature of the furnace. Combustion heat determinations were performed in a calorimetric bomb as per standard (ISO 1716). The test methodology assumes complete combustion of the fuel sample in an oxygen atmosphere under pressure, with ignition achieved by glowing of an electric spiral and measuring the temperature rise of the water in the calorimetric vessel. Thermogravimetric measurements were carried out using the dynamic method in accordance with the standard (EN ISO 11358-1:2014-09), recording changes in mass of the material subjected to the test, as a function of temperature, at a constant heating rate equalling to 10°C/min in an air atmosphere. The whole process is then recorded by the software and, once the test has been finished, the data obtained need to be described.

4. Results of testing

The results are presented in Tables 2 to 4. In studies concerning the determination of MITL as the ignition criterion, the appearance of embers in the heaped layers was observed both at a constant and at a constant increase in the temperature of the heated surface (Woźnica, 2021).

Table 2: Values of MITL at constant temperature or constant rise in heated surface temperature, MTCD, heat of combustion of sewage sludge and its mixtures with extinguishing powders

Designated parameters sewage	Test substance Sewage sludge dust	Sewage sludge + ABC Favorit 20 % wt.	Sewage sludge + D Favorit 20 % wt.
MITL at constant temperature in heated surface temperature, °C / time to the ignition, min	Layer thickness of the dust - 5 mm 270/10	350/8	300/7
MITL at constant rise temperature in heated surface temperature, °C / time to the ignition, min	300/85	----	-----
MITL at constant temperature in heated surface temperature, °C / time to the ignition, min	Layer thickness of the dust 12.5 mm 250/52	330/45	280/55
MITL at constant rise temperature in heated surface temperature, °C / time to the ignition, min	290/96	----	-----
MTCD, °C	450	430	420
Heat of combustion, MJ/kg	10.42	9.123	9.345

Table 3 presents experimental results of the determination of the spontaneous combustion temperature T_{Si} (hot storage) and the induction time t_i for sewage sludge dust samples (Woźnica, 2021).

Table 3: Summary of test results, TSi and ti determinations for sewage sludge dust

Sewage sludge dust:		
Volume of the measuring vessel (basket), cm^3	Self-ignition temperature T_{Si}, °C	Induction time t_i, h
21	158	0.9
50	148	1.8
100	144	2.4

Figure 1 presents one of experimental curve obtained during spontaneous combustion measurements for sewage sludge dust samples (Woźnica, 2021).

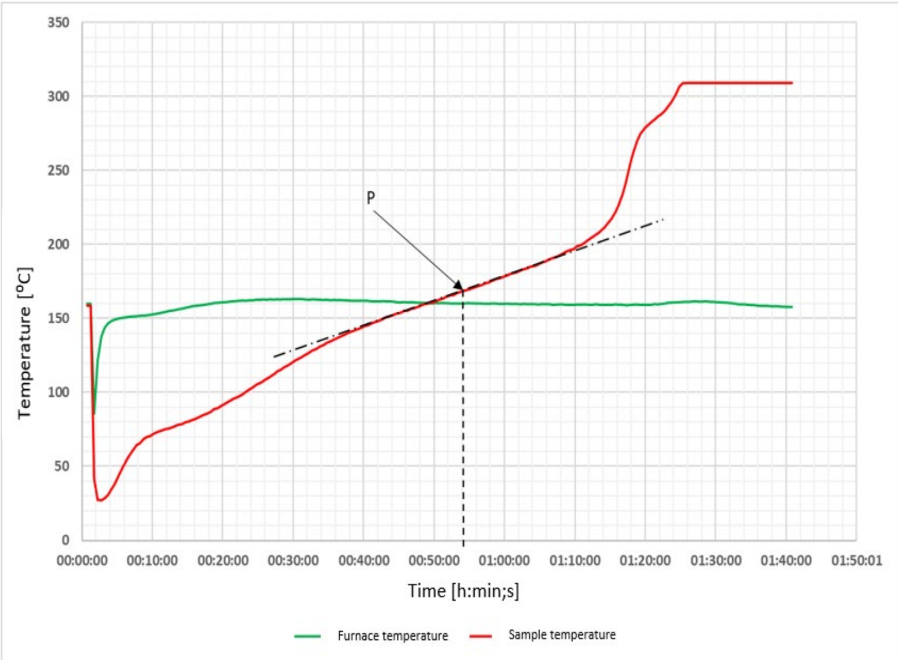

Figure 1: An example of a self-heating diagram of the tested sludge dust for a sample volume of 21 cm^3 and at a furnace temperature (hot storage in bulk) of 158 °C

During the heating of the sample in the temperature range from 20 °C to 800 °C a process of thermal degradation associated with mass loss takes place. Readings of characteristic parameters from thermogravimetric curves (TG, DTG curve) for sewage sludge are presented in Table 4.

Table 4: T Results of thermogravimetric analysis for sewage sludge dust

Designated TG parameters	Values
Temperature of thermal decomposition, °C	234
Temperature of 5% weight loss, °C	110
Temperature of 50% weight loss, ° C	423
Temperature of the end thermal decomposition, °C	749
Temperature of the maximum rate of weight loss, °C	272
Mass of the residue, %	38.84

5. Review of results

When analysing the test results it should be borne in mind that dewatered sewage sludge is a combustible material that has properties similar to those of organic combustible materials (biofuels). The combustion heat of dried sewage sludge is 10.42 MJ/kg, which is relatively low compared only to biofuels the energy value of which ranges from 8.00 to 25.00 MJ/kg. However, it is important to note that sewage sludge is a heterogeneous substance, composed of both flammable and non-flammable components, where their individual contribution determines the energy value. Taking into account the values of MITL for sludge dust, it can be stated that deposited layers of sludge dust (5 mm thick) on the hot surfaces of constant-temperature equipment (regular operating mode of the equipment) already at lower surface temperatures lead to the ignition of the layer (270°C) at much lower surface temperatures than during the increase in temperature of the furnace (e.g. due to equipment failure). The times until reaching of MITL were found to be several times higher if a constant temperature increment of the heated surface was applied. Higher MITL values were recorded when during the conducted tests dried sewage dust was used with firefighting powders. It was observed that the addition of 20 % by weight ABC Favorit fire extinguishing powder to the tested dried sewage dust was more effective than the addition of 20% by weight of D Favorit. The addition of ABC Favorit has increased the value of MITL for the 5 mm layer from 270°C to 350 °C. As regards D Favorit fire extinguishing powder, only an increase to 300°C, i.e. an increase by only 30 °C, has been recorded. The obtained results for ignition from the heated surface allow establishing the maximum permissible temperatures for devices working in dusty atmospheres according to the standard PN-EN 60079-14. The maximum allowable surface temperature of the device (MASTD) working in an atmosphere of surrounding sewage sludge dust (with the layer thickness being 5 mm) must not exceed 195 °C, and this value should be indicated on the rating plates of equipment operating in the presence of this dust in Ex zones. The addition of fire-extinguishing powders, especially ABC Favorit, was found to enhance fire safety endangered by ignition from a heated surface as the MASTD for a 5 mm layer is 80 °C higher than the MASTD of dried sewage dust. During testing executed pursuant to EN 15188, critical parameters for safe storage were determined for the tested dust, which were only fulfilled under the given conditions of safe storage of the sludge in the mass. Spontaneous combustion of the sludge occurred according to EN 15188 at the point of inflection. An increase in the volume of stored dried material from 21 to 100 cm^3 leads to a reduction in the spontaneous combustion temperature by 14 °C. Approximating these relations to a larger volume of dried material stored, e.g. for a 100 m^3 pile of sewage sludge, its spontaneous combustion may be expected to occur at the temperature of approximately 60 °C and in about one and a half months. The most intense pyrolysis process took place between 200-350 °C where the mass loss was almost 3 %/min and a high degree of conversion of the sample from solid to gas can be observed. In the second temperature range 400-490 °C it was slightly lower. This can be confirmed by the varied composition of the dried sewage. The residual mass is more than 1/3 of the whole sample, mainly metals, which are present in the sample.

6. Conclusion

Sewage sludge dusts are combustible and have relatively low ignition temperatures (below 300 °C) when deposited in layers on heated surfaces. The tested sludge may cause problems related to storage due to its ability to self-ignite, which generates additional problems with regard to the continuous control of the dried material in terms of temperature increase or compliance with the temperature range and volume in which it is stored in accordance with the obtained results. They have relatively low heat of combustion values for biomass. Thermogravimetric analyses indicate that the onset of pyrolysis occurs as early as at 234 °C, but produces about 40 % pyrolysis residues (most likely metals). The self-ignition temperature value depends on the volume of the stored material. For the biggest studied volume (100 cm3), the lowest self-ignition temperature was recorded which equalled to 144 °C. An analysis of the obtained test results allows the presumption that the dried sewage dust is a combustible material with properties similar to combustible materials of organic origin (biofuels). It was reported that 100 cm^3 of stored sludge undergoes spontaneous ignition at an oven temperature of 144 °C in an induction time of 2.4 hours. Taking into account the obtained results of the sludge dust with added extinguishing powders, it can be concluded that the extinguishing powders did not completely eliminate the risk of sludge ignition, yet increased the values of MITL accordingly. As a result of an analysis of the action of selected extinguishing powders on sludge by means of measurement of MITL and spontaneous combustion in the volume of mixtures of dust with extinguishing powders, it turned out that the least effective solution was the use of the extinguishing powder D Favorit. This powder is intended for extinguishing metal fires by forming an insulating layer. It proved to be less effective when mixed with flammable material. The most effective turned out to be ABC Favorit extinguishing powder, which is suitable for extinguishing both flame and non-flame fires, when the active substance was monoammonium phosphate.

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