Journal of Loss Prevention in the Process Industries 38 (2015) 1–10

Contents lists available at ScienceDirect

Journal of Loss Prevention in the Process Industries

journal homepage: www.elsevier.com/locate/jlp

Combining product engineering and inherent safety to improve the powder impregnation process

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ARTICLE INFO

Article history:
Received 26 June 2015
Received in revised form
18 August 2015
Accepted 18 August 2015
Available online 22 August 2015

Keywords:
Powder mixtures
Dust explosion
Textile
Flame retardant
Impregnation process
Polymer

ABSTRACT

The functionalization of nonwoven textiles can be realized by dry powder impregnation. In order to develop and improve this process, two complementary approaches have been combined: product engineering and inherent safety. It consists in integrating ab-initio consumers' requirements, production constraints as well as safety and environmental considerations. This case study is focused on the proposal, the characterization and the selection of powders mixtures of flame retardants and copolyesters, which will be used to create fire-proofed textiles. The influences of the chemical natures of the flame retardant (e.g. calcium carbonate, aluminium trihydroxide, ammonium polyphosphates), their respective concentrations, particle diameters and the addition of silica to flame retardant/polymer mixtures on their minimum ignition energy has been investigated. It has been determined that ammonium polyphosphates are far more efficient than other flame-retardants and that a minimum of 20%wt. concentration is needed to generate a powder mixture that will be almost insensitive to ignition by an electrostatic source. Modifying the particle size distribution and introducing glidants play also a significant role on flame retardant/polymer interactions, on powder dispersibility and has a strong impact on the minimum ignition energy. Finally, the formulations which have been selected fulfill the requirements for fire resistance, flowability, prevention of dust explosion; they are non-toxic, environmentally friendly and their cost is reduced.

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1. Introduction

Nowadays, it appears clearly that the existing process industries, in particular the high-added value industries, cannot simply evolve wondering ‘how to produce’ but should also take the societal, safety and environmental demands into account. It is even more obvious that the design of new processes must be underpinned both by obtaining functional properties and by integrating ‘ab initio’ safety and sustainable development requirements. Thus, combining product engineering and inherent safety approaches may be relevant. Moreover, the steps of product engineering and inherent safety procedures are often linked. For instance, in the four stages-procedure proposed by Moggridge and Cussler (2000) and detailed by Hill (2008), the modeling of the product properties, the selection of the product composition as well as the process design and risk assessment phases can be entirely shared by the inherent safety strategy (Kletz, 1998; Amyotte et al., 2009). More specifically, understanding and integrating the process–product interactions are essential to design a safer process as well as to obtain materials with desired features and functional properties for the consumers.

In order to illustrate the benefits of combining these complementary approaches, this article will be focused on the peculiar case of dry powder impregnation process. This activity especially concerns technical textiles used in transportation and building markets. To fulfill the specifications for such applications is not an easy task: the consumers are sensitive to design and comfort, but health, safety and environmental issues (as recycling) also have to be considered under strict regulations and, obviously, these requirements must be compatible with the fabrication process (as the powder flowability). Among all the textile materials that can be used are needled nonwoven polymers, including polyesters. These components are often flammable. Furthermore, European regulation insists on rethinking the materials used in internal upholstery to improve their recycling abilities. In order to fulfill these requirements, textile materials are often functionalized to modify their properties: nonwovens textiles can become conductive, fire-proofed, water repellent or antibacterial. They can also be coated,

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http://dx.doi.org/10.1016/j.jlp.2015.08.001
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printed, flocked or dyed and combined with other material to create composites.

Functionalization of textiles can notably be achieved by dry powder impregnation. This technology allows the incorporation of particles within the textile by applying a strong alternative electric field on both sides of the fibrous substrate (Marduel, 2012; Caramaro and Marduel, 2013). Initially, additives and thermoplastic powders are mixed and dispersed onto the textile surface. The impregnation level can be varied by modifying the residence time and the electric field. A 1–5 s residence time is sufficient to lead to a homogeneous distribution of the powder inside the substrate. In a last step, due to the presence of a thermoplastic material, the powder can be fixed within the textile by means of a heating post-treatment (Bonin and Ville, 2014).

This impregnation process combines combustible powders with heat and electrostatic fields, which raises questions about the ignition probability during manufacturing. Owing to the presence of polymers and electrostatic fields, a quantitative assessment of the dust explosion risk is then compulsory, especially as sparks and electrostatic discharges are identified as the most frequent ignition sources for plastic dust explosions (Van Laar, 1991; Beck et al., 1997; Glor, 2005; Janès and Carson, 2013; Janès et al., 2014). This risk assessment step will include the characterization of the pure powders and their mixtures, the determination of their ignition sensitivity but also the implementation of a risk management plan ensuring the process safety through preventive and protective barriers. At this point, combining product engineering and inherent safety becomes evident because one of the main requirements for such textiles is to be fireproofed. Thus, using flame retardant (FR) additives can both improve the process safety and ensure the functionalization of the final product. This action illustrates the moderation principle of inherent safety, i.e. combat risk at source using hazardous materials in their least hazardous forms (Kletz, 1977). The relevance of adding inert solid materials to combustible dusts to reduce explosion risk has already been pointed out by various authors (Amyotte et al., 1992; Bideau et al., 2011; Dufaud et al., 2012; Janès et al., 2014). Within the context of textile industry, the fillers or flame retardants have to be efficient to prevent ignition but should also be a substance with a low toxicity and ecotoxicity, be easily available, inexpensive and easy to recycle.

In this study, the ignition sensitivity - more exactly, the minimum ignition energy (MIE) - of thermoplastic powders, flame retardants and their mixtures has been investigated. Each compound or mixture has been characterized by their particle size distribution, densities, surface properties, thermal behavior (thermogravimetric analyses), rheological and electrostatic properties and homogeneity. The fire-resistance properties of the resulting textiles have also been determined with regard to the ignition sensitivity of the powders. In this article, we will mostly focus on the influence of various parameters on the minimum ignition energy of the powders used in this impregnation process: the chemical natures of the polymer and the flame retardant, their respective concentrations, particle diameters and the addition of silica to flame retardant/polymer mixtures. The impact of the mixing process has also been studied (chaotic stirrer, internal mixer or high shear granulator) but will not be developed here.

2. Generate mixtures to meet the needs

Moggridge and Cussler (2000) proposed a 4-steps procedure for chemical product design: (1) identification of customer needs, (2) generation of ideas to meet the needs, (3) selection of the ideas and (4) product manufacturing. The needs being defined, 'ideas' or proposals of powders mixtures have been 'generated' based on the previous requirements and on the characteristics of the pure compounds.

2.1. Binders, flame retardants and flowing agent

Three kinds of powders have been introduced in the mixtures: a thermoplastic polymer (binder), a flame retardant (FR) material and, as a function of the rheological properties, a flowing and anti-caking agent.

Tests were carried out on two epoxy-polyester polymers designated by the references: CoPE1 and CoPE2 respectively provided by Schaetti and Dupont coating. The chemical nature of the binders has been chosen with regard to their thermal characteristics (e.g. melting temperature), their affinity with the fibrous media, their rheological and tacking properties which are in accordance with the impregnation process. Due to their physico-chemical characteristics (Table 1) and their low cost, such polymer powders are commonly used in the textile industry.

One of the main requirements for the product is to be fireproofed. As a result, various flame-retardants have been tested as additives, while keeping in mind that they should be efficient but also that they should neither produce highly-toxic fumes nor pollute environment. Four distinct principles can be observed as a function of the nature of the flame-retardants. They can act as a heat-sink when their degradation is endothermic, dilute the oxidizing atmosphere by generating inert gases such as water, nitrogen or carbon dioxide, they can quench the radicals (e.g. hydroxyl radicals) or create a thermal insulation barrier in the solid phase (intumescence) (Janès et al., 2014). Radicals quenching is often obtained by using brominated or chlorinated compounds. However, their use does not meet the environmental and safety standards of the project. It should be also pointed out that introducing a non-combustible compound to a polymer will also automatically reduce the fuel content. As a consequence, three main categories of flame retardants have been selected: i) calcium carbonates, ii) aluminium trihydroxyde and iii) phosphorus based flame retardants.

Calcium carbonate, act both as heat-sink and gaseous phase inertant by decomposing with endothermicity and releasing carbon oxides. They are expected to have low environmental or human toxicities and are also rather inexpensive. However, as their effect is mostly based on their thermal properties and on their decomposition, relatively high loadings (Cashdollar and Hertzberg, 1987; Bartknecht, 1989; Eckhoff, 2003) are generally required to achieve a high efficiency and their volatilization should be fast, i.e. the residence time in the flame zone should be sufficiently long or the particle size has to be rather low (Sapko et al., 2000; Amyotte, 2006). Moreover, various authors have already investigated the effect of such mineral powders, especially limestone, on dust explosion inerting (Amyotte et al., 1992; Amyotte, 2006; Harris et al., 2010; Man and Teacoach, 2012). Their characteristics are listed in Table 1 (Minox1, Minox3 provided by Provencal and Minox2 provided by Merck).

Aluminium trihydroxyde (ATH) (ATH1 provided by Alteo) are often used as flame retardants. They also act as heat-sink and gaseous phase inertant as their thermal degradation is endothermic and releases water vapor. ATH is commonly used for fiber reinforced plastics and especially in medium and high voltage applications because they have very good thermo-mechanical and dielectric properties. They can be mixed with ammonium polyphosphate or phosphinate organic, which allows a significant reduction of the total flame-retardant ratio (Horrocks and Price, 2001; Hapuarachchi, 2009; Laoutid et al., 2009; Hull et al., 2011; Hollingbery and Hull, 2012; Naik et al., 2013; Zheng et al., 2014).

Phosphorus based agents are very efficient halogen free flame retardants that can be organic or mineral compounds. When

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