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ORIGINAL PAPER

Encapsulation of Oxidizers: Efficient Method by Spout-fluid Bed

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ABSTRACT: In composite solid propellants, the oxidizer in the form of particles is embedded in a polymeric matrix. In general, these oxidizers consist in inorganic salts that are hygroscopic, chemically incompatible or sensitive to friction or impact, so that microencapsulation can be applied as a mean to provide a protective coating layer. This work aims to assess the effectiveness of the spout-fluid bed method to perform microencapsulation of ammonium perchlorate particles with acrylic-based resin. The formed coating integrity was assessed by an optical stereomicroscope for samples with one, two and four layers of coating before and after dissolving the cores in water. The parameters utilized in this method provided a complete and individualized encapsulation with sufficient integrity. Therefore, the spout-fluid bed method proved to be effective, particularly with the application of multiple layers.

KEYWORDS: Ammonium perchlorates; Solid rocket propellants; Composite propellants; Encapsulating.

INTRODUCTION

Microencapsulation can be applied to coat particles for different purposes such as isolation, control of core release, safe handling, masking of a specific characteristic or to protect it from the external environment (Jyothi *et al*. 2010; Nagamachi *et al*. 2009). On the other hand, oxidizers particles in composite propellants are embedded in a polymeric binder that also serves as a fuel, and even though many inorganic compounds are categorized as such, ammonium perchlorate (AP) is by far the most employed oxidizer in this kind of propellant. The need for microencapsulation of oxidizers usually comes from their high hygroscopicity, lack of chemical compatibility or high sensitivity to impact or friction. The coating material is usually made out of a polymeric resin whose cure or hardening advances to the extent that agglomeration is prevented, which otherwise would have a negative impact on particles packing and the maximum solid loading as a consequence. The coating material needs indeed to be chemically compatible with the oxidizer and should not impair the final propellant which must be previously verified.

The spout-fluid bed is a microencapsulation method in which a resin is previously diluted in a solvent and sprayed onto the particles such as paint. The cure or hardening gradually takes place as the solvent evaporation proceeds (Altzibar *et al*. 2010; Heintz *et al*. 2012; Guignon *et al*. 2002). In that process, the particles at the bottom-center of the bed are spouted upwards by warm air

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pumped from the bottom and then fall sideward forming a fountain. The particles then move down through the fluid-bed and back to the bottom-center due to the conical base shape. The cycle repeats and creates an internal circulating movement. The resin diluted in a solvent is sprayed on the top of the fountain, scattering droplets over the surface of the emerging particles where it cures or hardens. In this work, microencapsulation of AP particles with the spout-fluid bed method was assessed in order to determine its effectiveness to coat AP particles. The integrity of the capsules was examined by means of an optical stereomicroscope by comparing micrographs before and after immersion of the microencapsulated particles in water.

MATERIALS AND METHODS

Materials: ammonium perchlorate (mean diameter = 420 μm) of AEQ as a core material; aqueous solution containing 30% R180W acrylic resin (Redelease), 0.6% red dye and 69.4% water in mass as spraying material.

Methods: microencapsulation of AP was performed in a VFC-LAB Micro of Freund-Vector. Initially, 30.0 g of AP was put inside the conical vessel and let warm air (~ 45 °C) spout upwards through the particles bed at the rate of 130 $L.min^{-1}$, then 1.5 mL of spraying material was applied over the top of the formed fountain. The coated particles were left circulating for 10 min in order to make sure that the whole solvent was eliminated. The application was performed one, two or four times consecutively under the same conditions depending on the number of coating layers (one, two or four, respectively). Capsule integrity: Morphology and integrity of the capsules were examined by means of a Discovery V12 optical stereomicroscope of Carl-Zeiss; micrographs were taken before and after the coated AP particles were immersed in water and stirred manually (at room temperature) to accelerate the core dissolution.

RESULTS AND DISCUSSION

The samples with one, two and four coating layers are shown in Fig. 1. The uniform coloration indicates that the samples did not present agglomerates, suggesting that the parameters and conditions adopted in this process were appropriate. It is noteworthy that the color becomes darker as the number of layers increases.

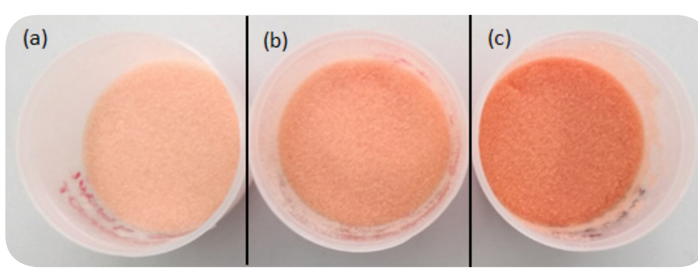

Figure 1. Samples of microencapsulated AP with acrylic-based coating: (a) one layer, (b) two layers and (c) four layers of coating.

The obtained microencapsulated AP was immersed in water in order to dissolve the core such that the capsule integrity could be better assessed, as shown in the micrographs in Figs. 2b,d. Figures 2a,c shows that the AP particles were uniformly microencapsulated, without evidence of any significant morphological changes or excessive accumulation of acrylic-based coating, which is seen in reddish color due to the presence of dye. The micrographs in Figs. 2b,d show the remaining acrylic-based coats after the AP core is completely dissolved in water. It is noteworthy that the remaining coatings in Fig. 2d (corresponding to the four-layer coating) preserved their morphology and structural integrity.

On the other hand, the remaining coatings morphology in Fig. 2b are not preserved, which suggests that the application of two layers is not sufficient to protect and preserve the capsules integrity. The remaining capsules in Figs. 2b,d had their reddish color faded, which is an indication of dye dissolution in water. The latter behavior has the potential to form emptiness or to cause flaws on the capsule that could lead to a lack of integrity, as seen in Fig. 2b:

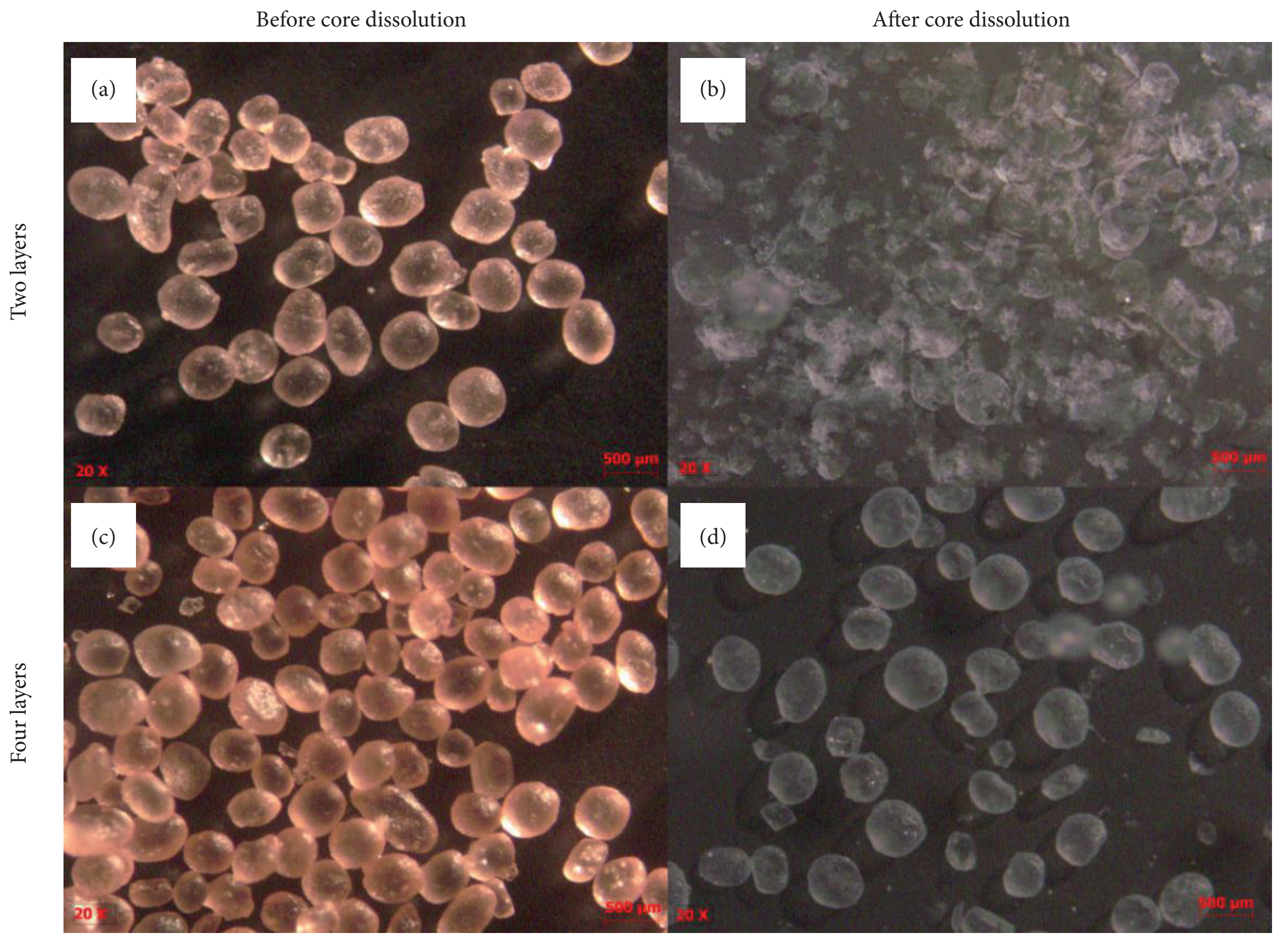

Figure 2. Microencapsulated ammonium perchlorate particles with acrylic-based coating: (a) two-layer coating before core dissolution, (b) two-layer coating after core dissolution, (c) four-layer coating before core dissolution and (d) four-layer coating after core dissolution.

CONCLUSION

This work has succeeded in assessing the spout-fluid bed microencapsulation method to apply acrylic-based coating on ammonium perchlorate particles. This method was capable to form individual and homogeneous capsules on AP particles surfaces with structural integrity, particularly, for microencapsulation performed with the application of multiple layers.

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AUTHORS' CONTRIBUTION

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