



All-polymer Layer by Layer coating as efficient solution to polyurethane foam flame retardancy

Federico Carosio^{a,*}, Claire Negrell-Guirao^b, Jenny Alongi^a, Ghislain David^b, Giovanni Camino^a

^a Dipartimento di Scienza Applicata e Tecnologia, Politecnico di Torino, sede di Alessandria, Viale Teresa Michel 5, 15121 Alessandria, Italy

^b Université de Montpellier II, Université de Montpellier I, Centre National de Recherche Scientifique and Ecole Nationale Supérieure de Chimie de Montpellier, IAM, Rue de l'école normale, 34296 Montpellier cedex 5, France

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ABSTRACT

This paper deals with the Layer by Layer assembly of an all-polymer chitosan/poly(allylamine diphosphonate) coating characterized by high efficiency in reducing the flammability of open cell polyurethane foams. The Layer by Layer assembly of the two components yields a supralinear growing coating that can be easily deposited on the complex 3D structure of polyurethane foams while preserving the open cell nature of the substrate. 2 bi-layers (BL) are enough to guarantee the deposition of a continuous thin coating capable of preventing the dangerous melt dripping phenomenon during flammability tests. The very same coating can dramatically reduce the heat release rate under a 35 kW/m² heat flux as it can efficiently hinder the foam structural collapsing during combustion. 2 BL coatings achieve a reduction of 48% while 4 BL reaches the maximum reduction of 55%, thus confirming the great efficiency of the coating in reducing the PU flammability at such low deposition steps and coating thicknesses.

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1. Introduction

During the last years, the Layer by Layer (LbL) technique has been presented and studied as a versatile surface engineering tool for conferring different kinds of properties to surface-based devices [1].

This technique, which fundamentals can be dated back to 1966, has been heavily exploited only after the reinvention by the group of Decher in early 90s [2,3]. One of the strong key parameters relies in the simplicity to step-by-step modify the surface of any materials, exploiting interactions occurring between the selected reagents [4]. The most common interaction is represented by the electrostatic attraction; indeed, the alternate adsorption of oppositely charged polyelectrolytes or nanoparticles, from water-based solutions/suspensions, allows for the build-up of stratified or highly interpenetrated nanostructured coatings [5]. The properties of these coatings can be controlled by finely tuning the deposition parameters such as molecular weight [6], temperature [7], ionic strength [8], and pH [9].

In recent years, the LbL has been used for the build-up of fire protecting coatings [10]. As recently reviewed, the LbL ability to modify the surface can play a key role when fire protection is needed. Indeed, during ignition and combustion of a material, the surface plays a predominant role as it basically controls mass and heat transfers between the flame and condensed phase. The heat reaching the surface is transmitted to the bulk, where volatiles that feed the flame by diffusing through the surface, are produced. Thus, engineering the surface via LbL allows for the control of the burning behavior of a polymer. The first LbL treatments aimed to fire protection were performed on cotton and synthetic fabrics [11–14]; then,

* Corresponding author.

E-mail address: federico.carosio@polito.it (F. Carosio).

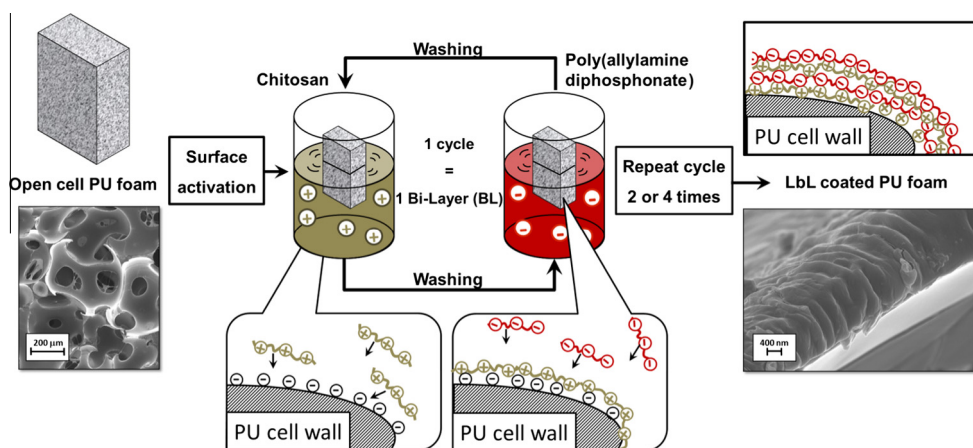


Fig. 1. Schematic representation of the adopted LbL process. PU foams are pre-activated by poly(acrylic acid) and then alternatively dipped in chitosan (positive) and poly(allylamine diphosphonate) (negative) solutions. The process is repeated 2 or 4 times in order to deposit 2 or 4 BL, respectively.

the technique has been extended to thin plastic films and foams [15,16]. These latter, especially in the form of open cell polyurethane (PU) foams, have been the subject of many flame retardant LbL studies during recent years [17–19]. Open cell PU foams pose the interesting feature of having a 3D structure that can be penetrated by the solution or suspension adopted in the LbL process thus depositing the coating on every surface available. Furthermore, foamed materials represent a severe fire-safety threat as they can easily catch fire, burn very quickly and exhibit melt dripping (i.e. formation of incandescent droplets of polymer) that can spread the fire to other ignitable materials [20].

The first attempt on PU foams has been made with polyelectrolyte stabilized carbon nanofibers; this LbL coating achieved a consistent reduction in the combustion kinetics, as assessed by cone calorimetry [15]. Subsequently clay nanoplatelets have been widely exploited and coupled with renewable polysaccharides or synthetic polyelectrolytes in LbL coatings [17,21,22]. Almost regardless of the adopted counterpart, all clay coatings managed to substantially reduce combustion kinetics and suppress the melt dripping, such results have been confirmed by real scale furniture mockup tests [22]. Coatings not containing nanoparticles have also been investigated showing that, if a high number of deposition steps is reached (i.e. 20), it is possible to achieve similar or better fire protection performances with respect of nanoparticle containing coatings [18,23].

Recent researches have been focused on the reduction of the required deposition steps in order to have substantial results. Up to now such reduction has been possible only for nanoparticle-based coatings; in detail, the presence of nanoparticles within the coating has been often referred as a mandatory condition in order to reduce the deposition steps while keeping the desired properties [24,25]. In this scenario, the production of all-polymer LbL coating with enhanced efficiency at low deposition steps represents a big scientific challenge. Furthermore, an all-polymer Layer by Layer coating would allow for the full exploitation of the tunability granted by polyelectrolytes and the possibility of a more conformal coating capable of adapting the complex 3 dimensional geometry of open cell polyurethane foams. Thus, in the present paper we are reporting an all-polymer LbL coating made with chitosan and poly(allylamine diphosphonate) characterized by high efficiency at low deposition steps. This LbL coating has been assembled on PU foams, following the scheme reported in Fig. 1.

The deposited chitosan/poly(allylamine diphosphonate) pair represents a system with enhanced char forming abilities. Indeed, chitosan is a polysaccharide which chemical structure may evolve toward thermally stable carbonaceous structures upon heating [26]; this char forming reaction can be further enhanced by the dehydration abilities of the poly(allylamine diphosphonate) [27,28].

The thermally stable structures, produced by the coating upon heating or flame exposure, can act as a physical barrier and protect the underlying polyurethane from heat and oxygen, thus resulting in a flame retardant effect.

Coating growth and morphology have been characterized by infrared spectroscopy and field-emission scanning electron microscopy. Thermal stability in inert and oxidative atmosphere has been evaluated by thermogravimetric analyses (TGA) while the flame retardancy has been tested by both flammability (application of a flame) and cone calorimetry (exposure to a heat flux). The deposition of only 2 BL of this all-polymer LbL system can uniformly coat every surface available on the PU foams, thus achieving a substantial reduction of the PU combustion kinetics (–50%) and, at the same time, suppressing the melt dripping phenomenon.

2. Experimental part

2.1. Materials

Open cell polyurethane foams with a density of 65 g/dm³ and a thickness of 15 mm were supplied by Compart (France). Branched poly(ethylene imine) (BPEI, Mw ~25,000 by Laser Scattering, Mn ~10,000 by Gel Permeation Chromatography, as

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