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Doctoral Dissertation  
Doctoral Program in Material Science and Technology (38<sup>th</sup> Cycle)

# **Polyelectrolyte complexes as building blocks for gas barrier coatings and fire-safe lightweight materials**

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Politecnico di Torino  
January, 2026

# Summary

This thesis deals with the use of polyelectrolyte complexation as an innovative approach for the development of functional materials through sustainable process. The work has been divided into two different topics: i) the deposition of sustainable gas barrier coating for packaging applications and ii) the production of fire-safe lightweight materials encompassing natural fibers from industrial wastes.

Within the first topic two different strategies were applied for the development of the coatings, the Layer-by-layer (LbL) assembly and the one step deposition of polyelectrolyte complex (PEC) on a PET film. Gelatin and montmorillonite were selected as building block for the coating and deposited onto PET substrate following surface activation by cold plasma treatment. Different morphologies were observed: a stratified, lamellar architecture for the LbL coatings and a homogeneous gelatin matrix embedding clay nanoplatelets for the PEC coatings. Oxygen barrier performances were evaluated by measuring oxygen transmission rates under dry and humid conditions. Both coating strategies led to a substantial reduction in oxygen permeability compared to pristine PET. The enhanced performance can be attributed to the formation of a “brick and mortar” structure that is able to increase the tortuosity path of permeating molecules.

Within the second topic, LbL and PEC assembly were employed for the fabrication of fire-safe lightweight composite materials with natural fibers recovered from tannery wastes. To this aim, three different assemblies were developed. In the first one, keratin fibers were surface coated using LbL deposition technique and then freeze-dried in order to obtain a porous material. The LbL coating based on chitosan (CH) and cellulose nanofibers (CNF) plays a crucial role in enabling the formation of self-supporting structures after freeze-drying. In the second assembly, gelatin and cellulose nanofibers were employed to form compact polyelectrolyte complexes (coPECs), which were combined with collagen fibers to prepare porous networks with different compositions and densities. The third assembly is composed by a gelatin/MXene matrix in which LbL-coated collagen fibers were dispersed in order to prepare a porous composite after freeze-drying. Composites in which the fibers were dispersed within a continuous matrix exhibited superior mechanical properties, as the matrix provided the primary contribution to the overall mechanical strength of the material. All prepared samples displayed

thermal conductivity values below 0.065 W/mK that is consider the common threshold for materials for insulation application. The incorporation of a relatively high (from 50 to 90%) fiber content produced a self-extinguishing behaviour during flammability tests in horizontal and vertical configurations. Moreover, forced combustion tests conducted at 50 kW/m<sup>2</sup> revealed low heat release rates and reduced total smoke production compared to values reported in the literature. In conclusion, the achieved results clearly show the potentialities of PECs either in delivering easy to upscale and high performing gas barrier coatings or in enabling the upcycling of fiber wastes into functional material characterized by excellent fire-safety and low environmental impact.