



Taylor-made aerogels through a freeze-drying process: economic assessment

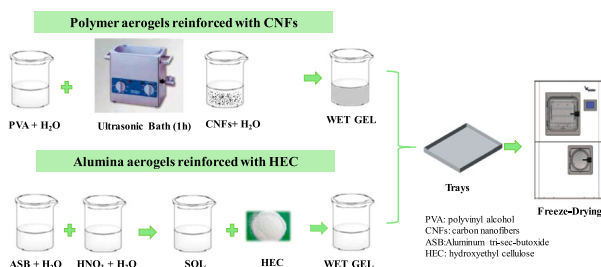
Carolina Simón-Herrero¹ · Amaya Romero¹ · Fernando Dorado¹ · Ignacio Gracia¹ · Jose Luis Valverde¹ · Luz Sanchez-Silva¹

Received: 20 September 2018 / Accepted: 2 November 2018 / Published online: 16 November 2018
© Springer Science+Business Media, LLC, part of Springer Nature 2018

Abstract

Polymer aerogels reinforced with carbon nanofibers and alumina aerogels reinforced with hydroxyethyl-cellulose have been successfully synthesized by means of a pilot plant freeze-drying process. Their main physicochemical properties have been measured and compared, and their production costs have been computed. The SWOT matrix of the process has been determined from internal and external analyses, revealing the interest of these products as building insulation materials and the need of establishing a detailed economic analysis. A homemade Excel-VBA application was designed in order to determine the economic parameters of the freeze-drying process. As a consequence of the total economic and physicochemical analysis, it was concluded that the production of aerogels reinforced with hydroxyethyl-cellulose could only be recommended if they are used as an insulating material in buildings with higher thermal stability requirements.

Graphical Abstract



Highlights

- Different types of aerogels have been successfully synthesised at pilot plant scale.
- Excel-VBA application was designed to determine the economic feasibility of the freeze-drying.
- Investment analysis and financial ratios demonstrated the interest of the polymeric aerogels.
- Aerogels reinforced with carbon nanofibers are recommended as insulating materials.

Keywords Freeze-drying · Economic analysis · Financial ratios · Building insulation

1 Introduction

The building industry is responsible for more than 40% of the total energy consumption in the European Union [1]. According to an International Energy Agency (IEA) analysis, energy consumption is predicted to be increased by 53% within the next 10 years as a consequence of the

✉ Luz Sanchez-Silva
Marialuz.sanchez@uclm.es

¹ Department of Chemical Engineering, University of Castilla La Mancha, Av. Camilo José Cela 12, 13071 Ciudad Real, Spain

economy and urbanization development [2, 3]. Therefore, energy conservation in the building sector by means of effective insulation strategies is crucial for solving the excessive energy consumption all over the world. Energy efficiency increase in buildings is an important goal for controlling energy demand [2], reducing the use of natural resources [3], and decreasing the greenhouse gases production [4]. Thus, the use of insulating materials in architectonic buildings is a key point to consider, but not the only one, since they can find applications in fire protection, personal comfort and condensation, and sound control [3].

Thermal insulators are composed by a material or composite materials with high thermal resistance, which decrease the heat flow rate by the mechanisms of conduction, convection, and radiation [3, 5]. In this sense, aerogels have gained importance due to their thermal insulation properties that would allow a significant increase of the energy efficiency in buildings. Aerogels are a solid derived from a gel in which the liquid compound has been replaced by a gas, thus leading to materials owning great porosity and, consequently, very light density [6–10]. The procedure used to dry the wet gel yielding the aerogel is the most important step of the whole process. Freeze-drying is recognized as the best method for solvent removal due to the high quality of the products obtained, keeping their residual humidity after drying below 5 wt% [11, 12]. Taking into account that the thermal conductivity of these materials increases with the moisture content [5], freeze-drying is considered to be a very competitive method to produce marketable insulating materials.

The main disadvantage of the commercialization of aerogels is their relatively low mechanical strength, which is a consequence of their high porosity. Organic aerogels are less crumbly and brittle than inorganic aerogels and present better thermal insulation properties than silica aerogels at ambient conditions [11]. Moreover, the polymer used for the synthesis of aerogels plays an important role in the properties of the ultimate products. Several studies have been focused on the preparation of organic aerogels from natural polysaccharides such as cellulose, and other polymeric matrix such as polyvinyl alcohol [10, 11, 13]. Furthermore, the addition of one or more reinforced materials to the polymeric matrix to improve the mechanical resistance of the aerogels is usually required [14–16].

This work is based on the synthesis of different types of aerogels (polymer aerogels reinforced with carbon nanofibers (CNFs) and alumina aerogels reinforced with hydroxyethyl-cellulose (HEC)) by means of a freeze-drying process performed at pilot plant scale. Apart from measuring their main physicochemical properties, this work explores the economic feasibility of producing aerogels from a freeze-drying process.

2 Methodology and plant description

2.1 Materials

2.1.1 Polymer aerogels reinforced with carbon nanofibers (CNFs)

Polyvinyl alcohol (10–98, Mw 61,000 g/mol) was supplied by Fluka Chemical Co. Ltd. Water was purified by distillation followed by deionization using ion exchange resins. CNFs were obtained according to the procedure described by Jimenez et al. [17] to be used as reinforcing material.

2.1.2 Alumina aerogels reinforced with hydroxyethyl-cellulose (HEC)

Aluminum tri-sec-butoxide (ASB, 97%), HEC (80–125 cp, 2% H₂O), and nitric acid (1 M) were supplied by Sigma-Aldrich Co., Ltd. and used as received. Nitric acid was dissolved in water to obtain a 0.2 M solution. Water was purified by distillation followed by deionization using ion-exchange resins.

2.2 Synthesis of aerogels

2.2.1 Polymer aerogels reinforced with carbon nanofibers (CNFs)

Polymer aerogels reinforced with CNFs were prepared by following a procedure based on that reported by Víctor-Román et al. [10]. A solution of polyvinyl alcohol in deionized water (2.5 wt%) was prepared by heating at 82 °C under vigorous stirring until its complete solution. Then, a certain amount of CNFs (0.25 wt% related to the polymer amount) was dispersed in deionized water using an ultrasonic bath (1 h at room temperature). After both solutions were cooled down to ambient temperature, they were mixed under vigorous stirring for 1 min to obtain the wet gel. The CNF suspension in the aqueous polymer solution was poured into the trays of the freeze-drying equipment (Lyobeta 6PL, Telstar Co. Ltd.). Once wet gel was introduced into the freeze-drying trays, it was frozen for 6 h at –60 °C and then sublimated under vacuum for 90 h.

2.2.2 Alumina aerogels reinforced with hydroxyethyl-cellulose (HEC)

Alumina aerogels reinforced with HEC were prepared by following a procedure based on that reported in previous works [14, 18]. A solution of ASB in deionized water with ASB/deionized H₂O molar ratio of 1/80, was prepared at 70 °C. Appropriate volumes of nitric acid (0.2 M) were added to the ASB solution to adjust the pH at 2–3. The solution

was then heated to 80 °C under vigorous stirring for 5 h to obtain the hydrosols. The proper amount of HEC (10 wt% related to the ASB solution amount) was then added to the hydrosol solution to obtain the wet gel. Once wet gel was introduced into the freeze-drying trays (Lyobeta 6PL, Telstar Co. Ltd.), it was frozen for 6 h at −60 °C and then sublimated under vacuum for 160 h.

2.3 Characterization of aerogels

The morphology of the aerogels was measured using a Phenom-ProX scanning electron microscope supplied by Phenom World. The pore size distribution was analyzed by means of a mercury porosimeter (Quantachrome Poremaster).

Thermogravimetric analysis (TGA) (Mettler Toledo TGA/DSC 1 STARe System) with a heating rate of 10 °C/min was carried out to perform the pyrolysis process and determine the thermal behavior of the materials.

Thermal conductivity of aerogels was measured using a single-needle (TR-1) sensor, in conjunction with the KD2 Pro Thermal Properties Analyzer (Decagon Devices). The Young's modulus was determined using dynamic mechanical analysis (DMA) 1 STARe System (Mettler Toledo), by means of stress–strain analysis in compression mode.

2.4 Economic analysis

The methodology of the economic analysis is shown in Fig. 1a. The first step in the economic analysis consists of analyzing the Strengths–Weaknesses–Opportunities–Threats (SWOT) matrix. SWOT matrix allows to explain the interest of the process (internal positive attributes and external attractive factors, strengths and opportunities, respectively) or the necessity to improve certain aspects of the process (internal aspects that are detracted from the process value and external factors that could be a risk for the process, weaknesses and threats, respectively). Once SWOT has been evaluated and the conclusion is determined to be positive, the investment analysis is carried out [10, 19].

In the present work, aerogels production using freeze-drying method has been selected as a model system for developing the economic analysis. The production per year was fixed in 186 m² of polymer aerogels reinforced with CNFs and 106 m² of alumina aerogels reinforced with HEC (both with 2.3 cm of thickness), as a consequence of the pilot plant capacity and the required time to obtain each product. The consumption of energy can be calculated from the power of the process. It was assumed a continuous operation process (8280 h of production per year).

2.5 Excel-VBA application: determination of the economic analysis of the freeze-drying process as a method to synthesize aerogels used as building insulation materials

A homemade Excel-VBA application was programmed to determine the economic parameters of the freeze-drying process to synthesize aerogels used as building insulation materials. First of all, Excel-VBA application analyzes the evolution of the NPV in the time horizon. In addition, the application allows to know the value of several economic ratios (ROE, EBITDA, IRR, Pay-back, Break-even-Point, credit worthiness margin of safety...) and their evolution during the time project. In order to determine the optimum selling price of aerogels, the Excel-VBA application analyzes the price curve, which allows to know for a specific value of the sales the Break-even-Point. Furthermore, the application allows to determine the sensitivity analysis to study the influence of possible divergences in the financial plan on the economy of the process (Fig. 1b).

3 Results and discussion

This work has been divided into four parts: the first part concerning the successful synthesis and characterization of aerogels in order to compare their physical and thermal properties. The second part is focused on the SWOT analysis of the freeze-drying process at pilot plant scale as drying method to dry the wet gel. The third part will allow to evaluate the equipment cost included in the investment analysis of the process to synthesize 2.16 m² of aerogels per batch. Finally, the last part of this work will establish a sensitivity analysis that allows to determine the influence of the different changes in the financial plan on the economy of the process.

3.1 Successful synthesis and characterization of aerogels

Polymer aerogels reinforced with CNFs and alumina aerogels reinforced with HEC were successfully obtained as reported in our previous work [10, 14]. Aerogels with undesirable characteristics were initially obtained. This fact was attributed to the freeze-drying conditions used. Operational variables such as freezing and drying time have a marked effect on the internal structure of the materials obtained [11, 20, 21]. Once aerogels were successfully obtained, they were characterized to compare their properties and verify that their use as building insulation materials in the architectonic buildings is feasible.

Table 1 summarizes the most important properties of the synthesized aerogels including SEM micrographs which allow to analyze the pore structure of the materials in detail.

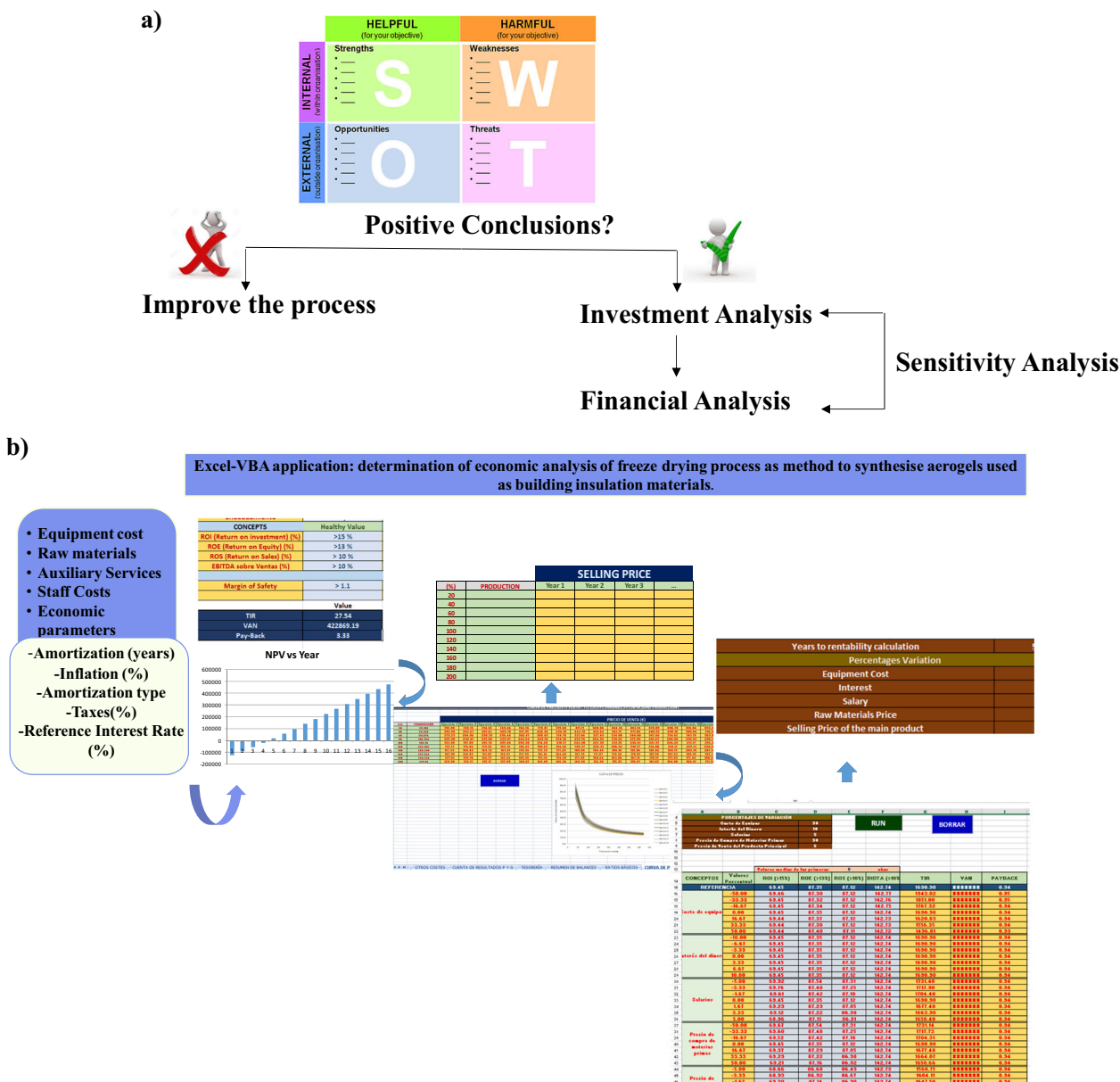
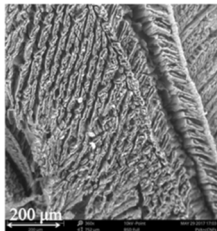
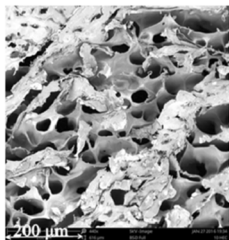
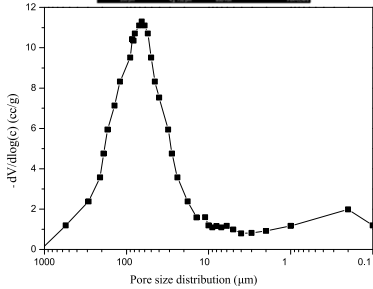
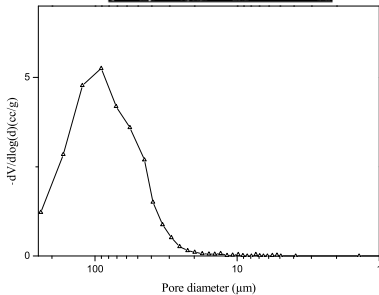
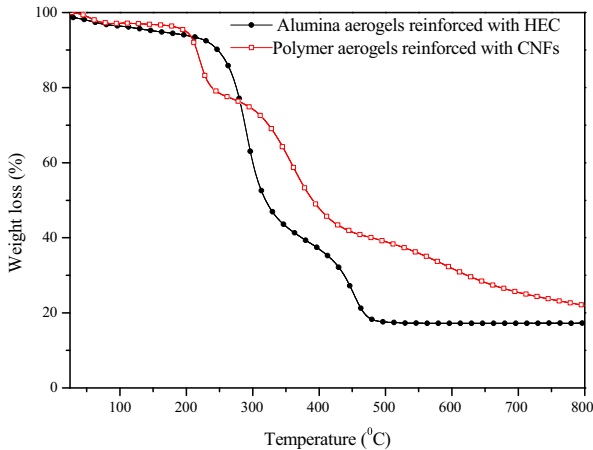


Table 1 Properties of aerogels synthesized by means of a freeze-drying process

	Polymer Aerogels reinforced with carbon nanofibers (CNFs)	Alumina Aerogels reinforced with hydroxyethyl-cellulose (HEC)
Physical Properties		
		
Young's Modulus (MPa)	0.77	0.58
Pyrolysis Analysis		
Thermal Properties		
	Thermal Conductivity (W/m·K)	
	0.0495±0.0284	0.0450±0.031

properties than alumina aerogels reinforced with HEC. This behavior was due to CNFs improved the mechanical properties in a higher extension than that of HEC fibers and other reinforcing agents as observed in other works [22].

3.2 SWOT analysis

SWOT matrix concerning the synthesis process of aerogels (Table 2) shows the strengths, weaknesses, opportunities, and threats that must be faced by the process. According to

Table 2 SWOT matrix for the production of aerogels by means of a freeze-drying process

	Strengths	Weaknesses
Internal analysis	<ul style="list-style-type: none"> -Value-added product: ultralight, insulating and good mechanical characteristics -Automatic equipment. -Wide market. -Cheap raw materials. -Low resource consumption. 	<ul style="list-style-type: none"> -Time consuming. -Low productivity. -No previous experience on trade.
External analysis	<p>Opportunities</p> <ul style="list-style-type: none"> -New synthesis route. -Unfulfilled customers. -Social awareness about responsible energy consumption. -Environment friendly. 	<p>Threats</p> <ul style="list-style-type: none"> -High cost investment. -Integration into the market. -Emergence of similar products or technology. -Social and economic instability.

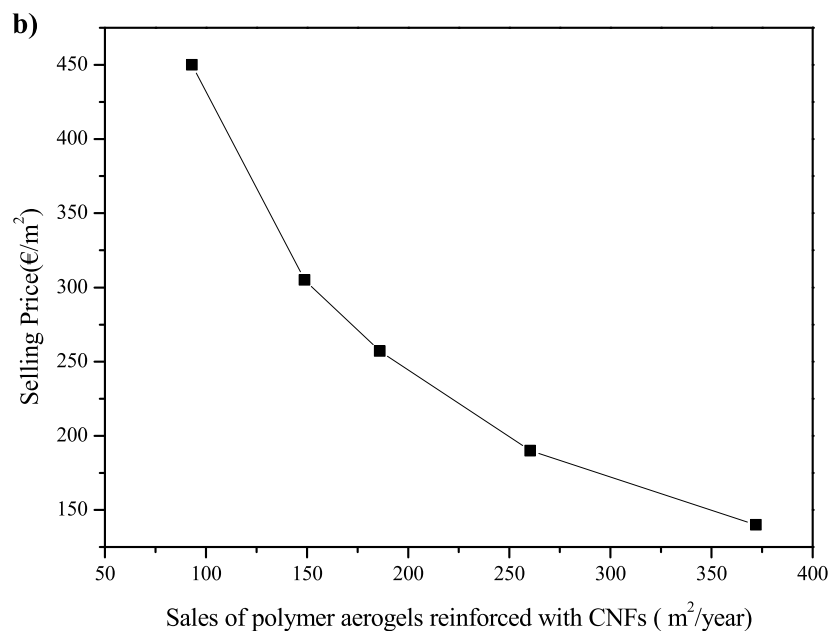
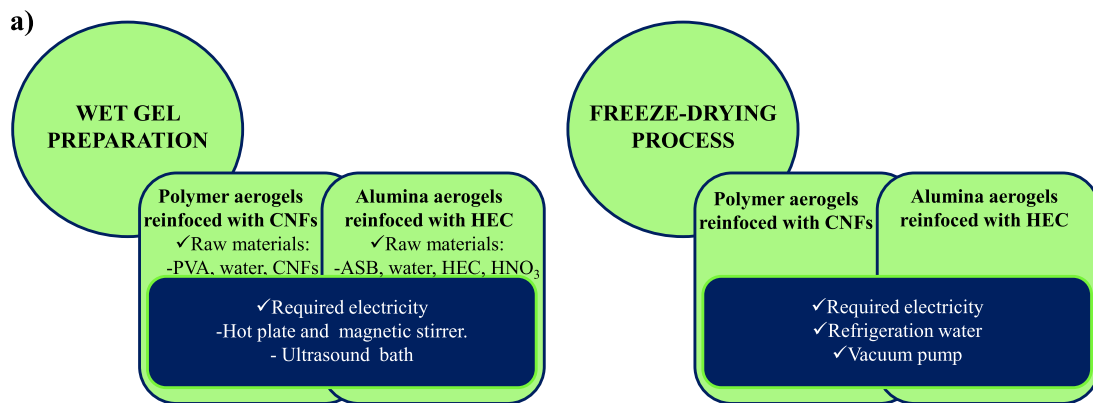


Fig. 2 a Raw materials, equipment, and utilities required in the synthesis of aerogels. **b** Price curve for polymer aerogels reinforced with carbonaceous nanomaterials by freeze-drying

Table 3 Equipment cost for aerogels production and working capital for the production of aerogels by means of a freeze-drying process

Equipment	Cost (€)	
Freeze-dryer Lyobeta 6 PL Telstar	84,281	
Ultrasound Bath Banderin Sonorix Digiplus	1720	
Hot plate and magnetic stirrer Agimatic-E, JP-Selecta	388	
Scenario 1	Raw material (€)	354
	Replacement (10%E) (€)	8639
	Total (€)	8993
Scenario 2	Raw material	6375
	Replacement (10%E)	8639
	Total (€)	15,014

SWOT analysis, the interest of the process can be explained with strengths and opportunities. Freeze-drying process is a novel drying method to get ultralights materials with high quality. This method allows to obtain dried products with very low values of residual humidity. Furthermore, this drying method is very competitive due to its environmental friendly characteristics. Besides, the know-how of this process provides the required information to characterize the final product. As it has been demonstrated in other works [11], the complete control of the variables of the process allows to determine the final characteristics of the materials. Thus, it is possible to adapt the process to the market demands.

The need to improve aspects of the process can be explained with weaknesses and threats. One of the most important threats is the high investment required to acquire the main equipment (lyophilizer). In addition, the production capacity of the freeze-drying pilot plant is low. As a whole, the global analysis of the SWOT matrix allows to get a real comprehension of the project situation.

3.3 Investment analysis

In this study, the investment analysis is based on a scale-up production of 2.16 m²/batch of polymer aerogels reinforced with CNFs and alumina aerogels reinforced with HEC (scenario 1 and scenario 2, respectively). Both scenarios require the aerogels manufacture to be integrated in a plant that is already operating for other purposes.

Figure 2a shows the raw materials, the equipment, and the utilities required in the different synthesis steps involved in the production of aerogels. Table 3 summarizes the cost of the equipment provided by the suppliers.

3.3.1 Capital

The capital is the investment required to start up the project and keep it active. It represents both the amount of money which must be supplied for the manufacturing and plant facilities, namely *fixed capital investment*, and that required for the operation of the plant, known as *working capital*.

The *fixed capital investment* was calculated according to the percentage method, which resulted to be 152,531€. However, some budget items were discarded since they are not necessary (for instance, the start-up of the lyophilizer because it is a turn-key setup). The percentage used should be determined on the basis of the type of process involved and the complexity.

Table 3 shows the *working capital* that was calculated as the raw materials stock for 1 month. Furthermore, the replacements were taken into account for the work capital estimation as a percentage of the total equipment cost (10%).

3.3.2 Sales and costs

The production capability of the pilot plant is 86 baths per year for scenario 1 and 49 baths per year for scenario 2 (345 working days per year). It is important to note that 249 working days have not been considered because the operator does not require more than 30% of his/her full working time to attend the whole process. Thus, the maximum annual production is estimated to be 186 and 106 m² for scenario 1 and scenario 2, respectively.

The market for this kind of materials reports different selling prices that vary depending on the final characteristics. Taking into account the list of prices of similar products developed by different companies (BuyAerogels, as an example), the typical selling price of aerogels in sheets ranges from 60 to 2100 €/m². Thus, after an exhaustive comparison of properties of commercial aerogels and those synthesized in this work, it will be assumed as a first approach to the economic analysis a selling price for the best aerogel made in this study of 500 €/m² (Table 4). Thus, considering that all the product was sold, the sales per year resulted to be 93 and 53 k€/year for scenario 1 and scenario 2, respectively.

Table 5 shows the amounts of raw materials, the price, and the suppliers required to obtain 2.16 m² of both types of aerogels (scenario 1 and scenario 2). The preparation of both types of aerogels requires the use of different equipment, which is associated with several electrical costs. These are lyophilizer, ultrasonic bath, and stirrer-hot plate. Table 6 summarizes for both scenarios the power and the operating time required to produce a batch (2.16 m²). As a result, the total cost of electricity per batch was 95€ for

Table 4 Commercial aerogels to be used as building insulation materials




Aerogel	Selling Price (€/m ²)	Thermal Conductivity (mW/m·K)	Maximum operating temperature(°C)	Reference
Airloy X103				
	2100	29	80	Buyaerogels
Airloy X134				
	2100	40.8	150	Buyaerogels
Spaceloft Blanket				
	200	14	200	Buyaerogels

Table 5 Amounts of raw materials, the price, and the suppliers required to obtain 2.16 m² of aerogels by means of a freeze-drying process

	Reactive	Mass (kg)	Supplier	Prize (€/kg)	Cost (€)
Scenario 1	PVA	0.54	Fluka Chemical	85.80	46.33
	Deionized water	21.6	University of Castilla-La Mancha	4.10·10 ⁻⁴	0.01
	CNFs	1.35·10 ⁻³	University of Castilla-La Mancha	600	0.81
Scenario 2	ASB	2.56	Sigma Aldrich	192	492
	HEC	3.85	Sigma Aldrich	139	535
	Deionized water	14.40	University of Castilla-La Mancha	4.10·10 ⁻⁴	6·10 ⁻³
	HNO ₃	32.4	Sigma Aldrich	17.5	567

scenario 1 and 164€ for scenario 2. Furthermore, water cost, as auxiliary service to be used for the freeze-dryer refrigeration, is also listed in Table 6, resulting in 0.62€ per batch for both scenarios.

Table 7 summarizes the total annual costs for the production of both types of aerogels, including staff costs (one plant employee). Directive personal costs have not been taken into account due to this pilot plant is integrated into a plant that should be already operating.

3.3.3 Income statement

The income statement shows the revenues, and the cost charged against these revenues, including depreciation, amortization, and taxes. Furthermore, investors could know if the project made or lost money during all the period.

Table 7 also shows the most relevant parameters used in the computation of the income statement. The time period used for the project was of 15 years due to it was not considered to be as a risky project. The investment curve is

Table 6 Power and the operating time to produce a batch (2.16 m²) of aerogels by means of a freeze-drying process

	Equipment	Power (kWh)	Time (min)	Supplier	Consumption (kWh)
Scenario 1	Hot plate and magnetic stirrer	0.50	1080	P-SELECTA Multimatic-9N	9
	Ultrasound batch	0.83	360	Banderin Sonorex Digiplus	4.9
	Freeze-Dryer. Freezing step	6.2	360	Lyoquest	37.2
	Freeze-Dryer. Drying step	9.0	5400	Lyoquest	810
Scenario 2	Hot plate and magnetic stirrer	0.50	1080	P-SELECTA Multimatic-9N	9.0
	Ultrasound batch	0.83	360	Banderin Sonorex Digiplus	4.9
	Freeze-Dryer. Freezing step	6.2	360	Lyoquest	37.2
	Freeze-Dryer. Drying step	9.0	9600	Lyoquest	1440
Refrigeration Water	Consumption (m³/h)		Price (€/kg)	Cost(€)	
	0.25		4.10·10 ⁻⁴	0.62	

Table 7 Total annual cost to produce 186 and 106 m² for scenario 1 and 2, respectively, and relevant economic parameters

Scenario 1	Raw material (€)	4055
	Auxiliar Services (€)	8200
	Staff Costs (€)	22,800
	TOTAL (€)	35,055
Scenario 2	Raw material (€)	78,100
	Auxiliar Services (€)	8100
	Staff Costs (€)	22,800
	TOTAL (€)	109,000
Amortization along (years)	15	
Inflation (%)	2	
Amortization	Linear, 15 years	
Taxes (%)	35	
Reference Interest rate (%)	3	

based on 100% in the start-up year and the amortization is considered linear during the whole time of the process. Furthermore, the working capital is recovered in its totally at the end of the project. Results for the considered period and for both possible scenarios are reported in Table 8.

Gross profit was calculated assuming that 100% of the total production was sold. The value of earnings before interest, taxes, depreciation, and amortization (EBITDA) showed a positive value (for scenario 1) over the accounting period, revealing the increase of the earnings over time. It was calculated by subtracting from gross profit only the

staff cost since general expenses (marketing, consulting, insurance, mailing, stationery...) were not considered in this project. EBIT (earnings before interest and taxes) was calculated by subtracting the amortization of the fixed capital investment from EBITDA. In this project, EBT (earnings before taxes) was the same than EBIT due to financing expenses were not applied. The project did not consider costs derived from external financing. The net profit, estimated by deducting the value of the income tax from EBT, showed increasing positive values over time for scenario 1. It was not possible to calculate income taxes for scenario 2 as a consequence of the profit absence.

Table 9 shows for scenario 1 the most representative financial ratios of the economic analysis over the accounting period. The value of the Return On Equity (ROE) shows the high capacity of the project to internally generate cash. Furthermore, the difference between the present value of cash inflows and the present value of cash outflows, NPV, resulted positive corroborating the economic viability of scenario 1. On the other hand, the Internal Rate of Return (IRR), which represents the reference interest rate that makes the NPV of all cash flows to be equal to zero, resulted to be 27.5%. Generally speaking, the higher the IRR of a project, the more the project profitability is. Finally, pay-back calculations for scenario 1 reported that a period of 4 years was needed to recover the investment made in the project. All parameters explained and shown in Table 9 demonstrated that the process to obtain polymer aerogels reinforced with CNFs at pilot plant scale (with a

Table 8 Income statement over the period

Financial year		2017	2018	2019	2020	...	2031
Scenario 1	Gross profit (€)	80,725	82,340	83,987	85,666	...	106,515
	Staff costs (€)	22,800	23,256	23,721	24,196	...	30,084
	EBITDA	56,922	58,057	59,214	60,394	...	75,040
	Amortization	10,169	10,169	10,169	10,169	...	10,169
	EBIT	46,754	47,888	49,045	50,225	...	64,871
	Financial expenses	–	–	–	–	...	–
	EBT	46,754	47,888	49,045	50,225	...	64,871
	Income taxes	14,026	14,366	14,713	15,067	...	19,461
Net income	32,728	33,522	34,331	35,157	...	45,409	
Scenario 2	Gross profit (€)	–33,285	–33,951	–34,630	–35,323	...	–43,919
	Staff costs (€)	22,800	23,256	23,721	24,196	...	30,084
	EBITDA	–56,085	–57,207	–58,351	–59,518	...	–74,004
	Amortization	10,169	10,169	10,169	10,169	...	10,169
	EBIT	–66,254	–67,376	–68,520	–69,687	...	–84,172
	Financial expenses	–	–	–	–	...	–
	EBT	–66,254	–67,376	–68,520	–69,687	...	–84,172
	Income taxes	–	–	–	–	...	–
Net income	–	–	–	–	...	–	

Table 9 Financial ratios

Financial year		2017	2018	2019	2020	...	2031
Scenario 1	ROE (%)	16.8	17.0	17.4	17.7	...	21.7
	NPV (€)	422,869					
	IRR (%)	27.54					
	Pay-back	Year 4					

ROE (return on equity): profit generated with the money that shareholders have invested; healthy value: >13%
 $ROE = \frac{\text{Net income}}{\text{Shareholders equity}}$

NPV (net present value): difference between the present value of cash inflows and the present value of cash outflows

$NPV = \sum_{i=1}^{i=n} \frac{C_i}{(1+r)^i} - C_0$; where C_i = net cash inflow during the period t ; C_0 = total initial investment costs; r = reference interest rate; n = number of time periods

IRR (internal rate of return): annual interest rate that makes the NPV of all cash flows from a particular project to be equal to zero

Table 10 Evolution of the pay-back and IRR as a function of the underestimation and overestimation of different parameters

			Pay-back	IRR
Scenario 1	Original situation		4	27.54
	Raw materials prize	+50%	4	26.61
		–50%	4	28.47
	Selling price of products	+50%	3	48.04
–50%		14	2.12	
Scenario 2 (1155 €/m²)	Original situation		13	3.23
	Raw materials prize	+50%	–	–
		–50%	5	23.99
	Selling price of products	+50%	4	33.67
–50%		–	–	

production of 186 m² of aerogels layers per year) was economically viable.

In order to determine the optimum selling price by considering a possible trend of the market, the price curve for scenario 1 was determined in a range of production between 172 and 43 batches/year (372 and 93 m² of aerogel per year) (Fig. 2b). The resulting curve allowed to know, for a specific value of the sales, the Break-even-Point, which represents the point from which the project begins to make profits. According to Fig. 2b, the higher the sales of aerogels, the lower the price was. Since a selling price of 500 €/m² was initially assumed for the product, 257 €/m² would assure the economic feasibility of the process (186 m²/year).

3.4 Sensitivity analysis

A sensitivity analysis was carried out to study the influence of possible divergences in the financial plan on the economy of the process. Thus, two parameters: raw materials price and selling price, were considered assuming overestimation and underestimations of 50% for both parameters (Table 10).

Regarding scenario 1, raw materials price variations did not influence considerably on the pay-back and IRR values. However, selling price influenced on the process economy. Results indicated that the higher the selling price, the lower pay-back value and the higher IRR values were. Underestimations of 50% of the selling price changed significantly the evolution of IRR and pay-back parameters resulting in non-profitability of the process.

As above mentioned, scenario 2 resulted to be the most critical one. After an exhaustive study of the economy of the process for the manufacture of alumina aerogels reinforced with HEC, the Break-even-Point resulted to be 1155 €/m², much higher than that obtained with scenario 1. The parameters considered for sensitive analysis influenced considerably on the process economy. Analysis indicated that the lower the raw materials price, the higher the IRR values were. As commented for scenario 1, pay-back decreases and IRR increases with increasing selling prices.

Summarizing, the synthesis of polymer aerogels reinforced with CNFs at pilot plant scale (with a production of 186 m² of aerogels per year) resulted in a very competitive process with good financial feasibility and viability. However, the synthesis of alumina aerogels reinforced with HEC was not economically viable due to several parameters such as the long time required for the production, the low production capability per year, and the high prices and amount of the raw materials. Production and commercialization of the alumina aerogels reinforced with HEC should be only recommended for building applications requiring higher thermal stability at temperatures below 280 °C.

4 Conclusions

Different types of aerogels (polymer aerogels reinforced with CNFs and alumina aerogels reinforced with HEC) have been successfully synthesized by means of a pilot plant scale freeze-drying process. Polymer aerogels reinforced with CNFs showed a lamellar porous structure while alumina aerogels reinforced with HEC showed homogeneous spherical pores. Moreover, the second decomposition stage of alumina aerogels reinforced with HEC shifted to higher temperatures in comparison to polymer aerogels reinforced with CNFs.

Results demonstrated the economic feasibility of the freeze-drying process to obtain polymer aerogels reinforced with CNFs. The investment analysis and the financial ratios demonstrated the interest of the project to product polymer aerogels reinforced with CNFs, and the price curve established the amount of sales required to satisfy the Break-even-Point. However, the synthesis of alumina aerogels reinforced with HEC was not economically viable considering a production of 49 batch/year and a selling price of 500 €/m². The Break-even-Point of alumina aerogels reinforced with HEC production was too high (1155 €/m²). Thus, the production of aerogels reinforced with HEC should be only recommended as insulating materials for buildings with higher thermal stability requirements.

Acknowledgements The present work was performed within the framework of the NANOLEAP project. This project has received funding

from the European Union's Horizon 2020 research and innovation program under Grant Agreement No. 646397.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Disclaimer We must indicate that this is an authors' original work, which has not been published elsewhere and is not under consideration for publication. The content of this work is approved by all the authors.

References

- Sierra-Perez J, Boschmonart-Rives J, Gabarrell X (2016) Environmental assessment of façade-building systems and thermal insulation materials for different climatic conditions. *J Clean Prod* 113:102–113
- Zhang T, Tan Y, Yang H, Zhang X (2016) The application of air layers in building envelopes: a review. *Appl Energy* 165:707–734
- Aditya L, Mahlia TMI, Rismanchi B, Ng HM, Hasan MH, Metselaar HSC, Muraza O, Aditya HB (2017) A review on insulation materials for energy conservation in buildings. *Renew Sustain Energy Rev* 73:1352–1365
- Dixon G, Abdel-Salam T, Kauffmann P (2010) Evaluation of the effectiveness of an energy efficiency program for new home construction in eastern North Carolina. *Energy* 35:1491–1496
- Al-Homoud MS (2005) Performance characteristics and practical applications of common building thermal insulation materials. *Build Environ* 40:353–366
- Cao F, Ren L, Li X (2015) Synthesis of high strength monolithic alumina aerogels at ambient pressure. *RSC Adv* 5:18025–18028
- He S, Zhang Y, Shi X, Bi Y, Luo X, Zhang L (2015) Rapid and facile synthesis of a low-cost monolithic polyamide aerogel via sol-gel technology. *Mater Lett* 144:82–84
- Han Y, Zhang X, Wu X, Lu C (2015) Flame retardant, heat insulating cellulose aerogels from waste cotton fabrics by in situ formation of magnesium hydroxide nanoparticles in cellulose gel nanostructures. *ACS Sustain Chem Eng* 3:1853–1859
- Nemoto J, Saito T, Isogai A (2015) Simple freeze-drying procedure for producing nanocellulose aerogel-containing, high-performance air filters. *ACS Appl Mater Interfaces* 7:19809–19815
- Víctor-Román S, Simón-Herrero C, Romero A, Gracia I, Valverde JL, Sánchez-Silva L (2015) CNF-reinforced polymer aerogels: influence of the synthesis variables and economic evaluation. *Chem Eng J* 262:691–701
- Simón-Herrero C, Caminero-Huertas S, Romero A, Valverde JL, Sánchez-Silva L (2016) Effects of freeze-drying conditions on aerogels properties. *J Mater Sci* 51:8977–8985
- Ratti C (2012) Handbook of food process design, freeze-drying process design. Wiley-Blackwell, Oxford
- Sánchez-Silva L, Víctor-Román S, Romero A, Gracia I, Valverde JL (2014) Tailor-made aerogels based on carbon nanofibers by freeze-drying. *Sci Adv Mater* 6:665–673
- Simón-Herrero C, Romero A, Valverde JL, Sánchez-Silva L (2017) Hydroxyethyl cellulose/alumina-based aerogels as lightweight insulating materials with high mechanical strength. *J Mater Sci* 53(1):1556–1567
- Eichhorn SJ, Dufresne A, Aranguren M, Marcovich NE, Capadona JR, Rowan SJ, Weder C, Thielemans W, Roman M, Renneckar S, Gindl W, Veigel S, Keckes J, Yano H, Abe K, Nogi M, Nakagaito AN, Mangalam A, Simonsen J, Benight AS,

- Bismarck A, Berglund LA, Peijs T (2010) Review: current international research into cellulose nanofibers and nanocomposites. *J Mater Sci* 45:1–33
16. Moon RJ, Martini A, Nairn J, Simonsen J, Youngblood J (2011) Cellulose nanomaterials review: structure, properties and nanocomposites. *Chem Soc Rev* 40:3941–3994
 17. Jimenez V, Nieto-Marquez A, Díaz JA, Romero R, Sánchez P, Valverde JL, Romero A (2009) Pilot plant scale study of the influence of the operating conditions in the production of carbon nanofibers. *Ind Eng Chem Res* 48:8407–8417
 18. He F, Sui C, He X, Li M (2015) Facile synthesis of strong alumina–cellulose aerogels by a freeze-drying method. *Mater Lett* 152:9–12
 19. Fernández-Ronco MP, de Lucas A, Rodríguez JF, García MT, Gracia I (2013) New considerations in the economic evaluation of supercritical processes: separation of bioactive compounds from multicomponent mixtures. *J Supercrit Fluid* 79:345–355
 20. Patapoff TW, Overcashier DE (2002) The importance of freezing on lyophilization cycle development. *BioPharm* 15(3):16–21
 21. Wang Y, Gawryla MD, Schiraldi DA (2013) Effects of freezing conditions on the morphology and mechanical properties of clay and polymer/clay aerogels. *J Appl Polym Sci* 129(3):1637–1641
 22. Simón-Herrero C, Gómez-Daza L, Romero A, Valverde JL, Sánchez-Silva L (2018) Nanoclay-based PVA aerogels: synthesis and characterization. *Ind Eng Chem Res* 57(18):6218–6225