Optimization of spray polyurethane foam systems for enhanced performance and cost efficiency

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Abstract

This project focuses on the development and optimization of spray polyurethane systems for building applications, carried out in Plixxent S.L., with the dual aim of improving performance and reducing formulation costs while complying with CE marking requirements. Four main systems were addressed: BA-WE2; BA-RF2, which was developed from the former; BA-RF1; and W-M, a system designed for mixed applications.

BA-RF2 was formulated by adjusting polyol ratios and blowing agents to create a cost-efficient alternative for roofing and flooring, while ensuring properties such as reactivity, dimensional stability, compressive strength, thermal conductivity and fire resistance. Experiments were conducted using the BA-WE2 system in order to adapt it for use with a co-blowing agent and achieve cost reduction. In parallel, modifications were made to W-M to enhance its thermal and mechanical performance and achieve a cost-effective alternative.

Two systems underwent experimental testing, BA-RF1 and BA-WE2, to verify their compliance with the technical requirements defined in EN 14315-1:2013. The results confirmed that the developed formulations meet the performance thresholds for their intended uses, demonstrating the feasibility of formulation adjustment as a tool for cost-effective and application-specific polyurethane design.

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1. Objective

The objective of this project is to develop and optimize spray polyurethane systems through experimental formulation and testing, in order to meet the technical and economic requirements of specific construction applications. The study pursues three main goals:

- To create a new formulation, BA-RF2, by modifying the wall-oriented system BA-WE2, as an alternative to the system BA-RF1, aiming to reduce raw material cost while ensuring the necessary performance for roofing and flooring applications in terms of reactivity, density, dimensional stability, thermal conductivity, compressive strength, and fire behaviour.
- To adapt BA-WE2 for its use with a co-blowing agent and achieve cost reduction.
- To improve the existing system, W-M, intended for mixed applications, by adjusting its composition to enhance thermal and mechanical performance while reducing formulation costs.

Additionally, two systems are subjected to experimental trials to verify their compliance with CE marking and the performance criteria established in EN 14315-1:2013.

2. Introduction

Polyurethane foam (PU) is a versatile and widely used polymeric material, known for its lightweight nature, insulating properties and adaptability. It was first developed in the late 1930s in Germany, by Dr. Otto Bayer and his team. They discovered the polyurethane reaction while searching for alternatives to rubber. In the 1950s, the material began to see widespread use in the market, as it evolved very rapidly into many applications across various industries, from construction and automotive to furniture and packaging.

This material can be broadly categorized into two main types, based on its structure and performance characteristics: flexible and rigid foam. Flexible polyurethane is soft, elastic, and commonly used in cushioning applications such as mattresses, car seats, and upholstered furniture. Rigid polyurethane usually provides excellent structural and thermal insulation, which makes it ideal for use in building panels, refrigeration units, pipeline insulation, cold storage facilities, among other applications. And with particular relevance to the focus of this work, its uses also include spray-applied insulation systems in construction (such as walls, roofs, floors, attics, and crawl spaces), where it plays a crucial role in enhancing energy efficiency, improving moisture control, and reducing air leakage.

Further distinctions can be made, based on specific properties, like cell structure (particularly the ratio of open-cell and closed-cell, which is crucial in spray systems), density, and fire resistance. These properties tailor the different types of foam to suit a wide array of needs.

Spray polyurethane foam (hereinafter referred to as SPF throughout this work for the sake of brevity and clarity), has a relevant place in the market, particularly in construction and insulation. It is applied as a liquid that rapidly expands into a solid foam that serves as an airtight barrier with superior thermal resistance. In the case of closed-cell systems, it provides high thermal and moisture resistance. Its characteristics and chemical complexity offer a wide range of possibilities and opportunities for improvement, in order to provide alternatives to modern materials engineering and energy-efficient design.

3. Theoretical background

The following theoretical background is primarily based on the works of Uhlig and Oertel [1,2], which provide comprehensive descriptions of polyurethane chemistry.

Polyurethane polymers are produced through the reaction between polyfunctional polyols and isocyanates, resulting in the formation of urethane linkage units, which is formed through the nucleophilic addition of an hydroxyl group of the polyol to an isocyanate group.

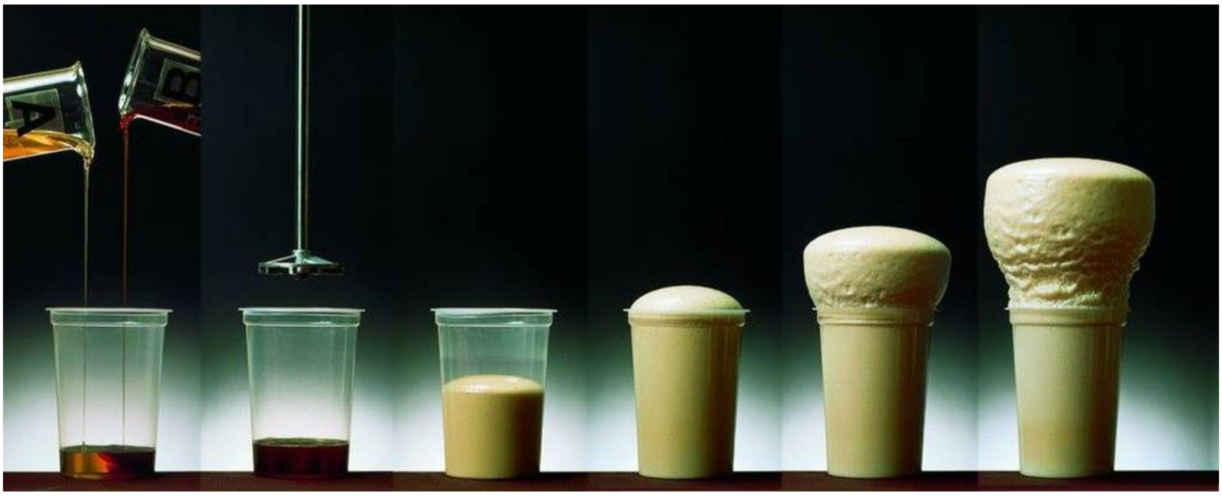


Figure 1. Polyurethane's free rise [3]

This reaction can be catalysed by organic amines or metallic compounds to control the reaction kinetics, additionally silicones, surfactants and other types of additives such as flame retardants and blowing agents, are employed to modify the final properties of the produced polyurethane foam.

3.1. Synthesis of polyurethane

Polyurethanes are formed through an addition polymerization mechanism, involving successive reactions of bifunctional or polyfunctional monomers (polyols and isocyanates), to yield high molecular weight polymers through the continuous formation of urethane linkages.

At molecular level the process begins with the nucleophilic attack of the hydroxyl group of the polyol to the carbon atom of the isocyanate group, which is highly electrophilic due to the electron-withdrawing effects of both the nitrogen and the oxygen atoms that are adjacent to it. Then a tetrahedral intermediate is formed, that eventually rearranges leading to the formation of the urethane linkage, completing the reaction.

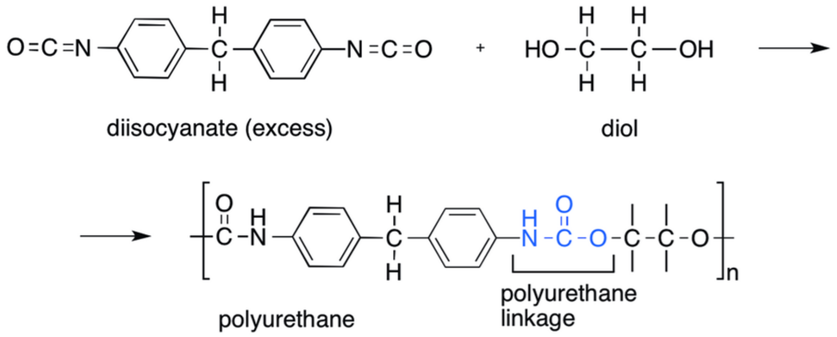


Figure 2. Reaction between a diol and a diisocyanate [4]

Because of the bifunctionality or polyfunctionality of polyols and isocyanates, the process repeats at multiple sites, which leads to the formation of crosslinked and linear polymers, depending on the functionality and ratios between components used.

3.2.Raw materials for polyurethane synthesis

3.2.1. Polyols

Compounds with multiple hydroxyl groups in the molecule are referred to as polyols. They dictate not only the reactivity of the system but also the mechanical, thermal and morphological properties of the final polymer. High molecular weight polyols (with average molecular weights up to $8 \cdot 10^3$ suppose the basis for the formation of the foam. Low molecular weight compounds, such as ethylene glycol, butanediol and glycerine, instead act as chain extenders or crosslinkers.

High molecular weight polyols can be classified into several types depending on their backbone structure and functional groups, the most relevant of which for spray polyurethane systems are the polyether and the polyesters.

3.2.1.1. Polyether polyols

Commonly used in the manufacture of rigid polyurethane foams tend to have a functionality of 4 or more. They are formed through the polyaddition of basic structural units. Monomeric epoxides are very used in this case because its ring is easily cleaved due to the high ring stress and the polar character of the bonds.

The use of this type of polyols implies more catalysis than with polyesters, due to its reduced reactivity in front of isocyanates. They present a very good hydrolytic stability and its use is advantageous in terms of viscosity, which remains not too high taking into account its functionality.

Aminic polyols constitute a group of reactive polyether polyols that contain primary or secondary amine groups, effective in the acceleration of the reactivity, also able to modify the morphology of the foam. They are characterized by containing both the hydroxyl and amine groups, which react with isocyanates forming urethane and urea linkages respectively.

Its high reactivity helps with faster tack-free times, very useful in SPF systems. It also contributes to network branching and higher crosslinking degree.

The amine-functionalized sucrose or Mannich base derivatives are two examples, last ones being very important for flame retardancy and improved thermal stability.

3.2.1.2. Polyester polyols

Formed by polycondensation between alcohols or phenols and carboxylic acids or anhydrides, such as ethylene glycol, glycerol, and on the other hand phthalic anhydride, adipic acid or maleic acid. During its production water has to be removed as it is released in the reaction.

They present higher polarity and greater hydrogen bonding capacity than polyether polyols. They contribute more to rigidity, being very useful in high-performance SPF formulations that require a high compressive strength and dimensional stability. Aromatic polyester polyols are also very useful by contributing to flame suppression during foam combustion.

Its main drawback is the high viscosity they confer to the polyol system, which may difficult the processing during spray application.

3.2.2. Isocyanates

Isocyanates are highly reactive compounds characterized by the presence of the isocyanate functional groups (-N=C=O). These compounds are fundamental building blocks in the production of polyurethanes, due to its reactivity and versatility. They react with alcohols, amines and water, forming several products.

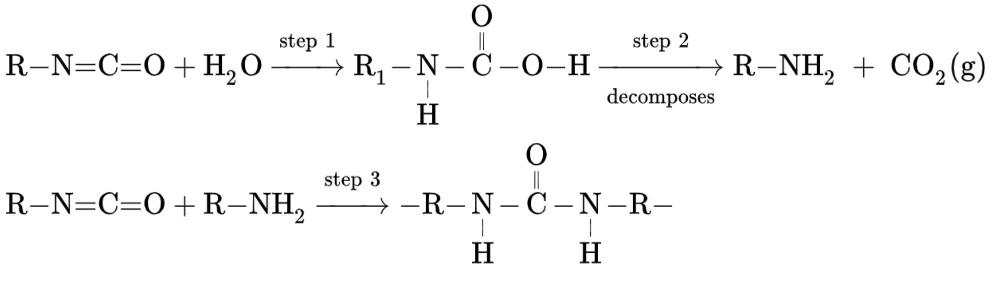
3.2.2.1. Reactions of isocyanates

The primary reaction of interest in polyurethane chemistry is the formation of urethane linkages, which takes place between isocyanates and alcohols in an exothermic process. This transformation constitutes the backbone of polyurethane polymer networks.

Additionally, isocyanates can react with primary and secondary amines, forming urea linkages that enhance the mechanical rigidity and thermal stability of the final polymer. This reaction is faster than with hydroxyl groups.

The other secondary reaction that is key in polyurethane chemistry takes place between an isocyanate and water. First of all, carbamic acid is formed. However, it is unstable and decomposes almost immediately, forming an amine and carbon dioxide gas.

As the amine is very reactive, quickly reacts with another isocyanate molecule forming an urea bond.



$$\mathrm{R{-}N{=}C{=}O} + \mathrm{H_2O} \xrightarrow{\text{step 1}} \mathrm{R_1} - \underset{\mathrm{H}}{\underset{|}{\mathrm{N}}} - \overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}} - \mathrm{O{-}H} \xrightarrow[\text{decomposes}]{\text{step 2}} \mathrm{R{-}NH_2} + \mathrm{CO_2(g)}$$

$$\mathrm{R{-}N{=}C{=}O} + \mathrm{R{-}NH_2} \xrightarrow{\text{step 3}} -\mathrm{R} - \underset{\mathrm{H}}{\underset{|}{\mathrm{N}}} - \overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}} - \underset{\mathrm{H}}{\underset{|}{\mathrm{N}}} - \mathrm{R} -$$

Figure 3. Reaction of isocyanate with water and with an amine [5]

This reaction is critical in foam production, because CO_2 acts as a chemical blowing agent, creating the foams characteristic structure. Polyurethane foam has a cell structure composed of gas-filled cells, which are formed during the foaming reaction. These cells can be either open or closed, depending on the formulation and processing conditions, and their morphology plays a key role in determining the material's thermal and mechanical performance.

It is also important to mention the reaction that occurs when isocyanate groups react with themselves through a trimerization mechanism, leading to the formation of polyisocyanurate rings, known as PIR. This reaction is catalysed by specific catalysts (often alkali metal carboxylates or amine-based systems). This linkages exhibit superior thermal stability, chemical resistance and rigidity compared to traditional urethane or urea bonds. The foam produced that has a remarkable presence of this molecules is often known as PIR foam.

3.2.2.2. Types of isocyanates

Since the reactivity of isocyanates is influenced by both electronic and steric factors, several types are used depending on the desired properties of the final products. Aromatic isocyanates (due to its electron-withdrawing nature of the aryl ring), generally exhibit higher reactivity than aliphatic isocyanates. Also the position and the number of isocyanate groups in the molecule affect the crosslinking potential, which in turns affects the rigidity of the polymer.

Methylene diphenyl diisocyanate (MDI) and its polymeric form (PMDI), are the most widely used in rigid polyurethane chemistry:

- MDI, on the other hand, is the most important type of isocyanate for rigid polyurethane. Its basic structure consists of two isocyanate groups, that are attached to a methylene-bridged diphenyl core. The rigid aromatic backbone of MDI is key to provide excellent mechanical and thermal properties. Its use is ideal for insulation foams, structural panels, and high-strength composites.

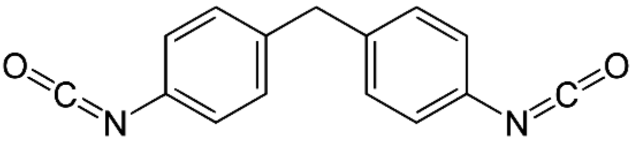


Figure 4. 4,4-MDI molecule [6]

- PMDI is the most commonly used form in rigid polyurethane foam formulations. It is a complex mixture that not only contains pure MDI (monomeric) but also oligomeric species that provide higher functionalities which enable a greater degree of crosslinking, that lead to rigid, thermosetting networks with a good dimensional stability and low thermal conductivity. In the present study, a product with an isocyanate content of about 31-32% of NCO and a viscosity profile of approximately 200 mPa·s at 25ºC is used.

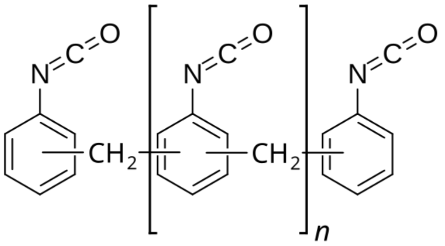


Figure 5. PMDI example [6]

3.2.3. Additives

When creating a system of polyurethane foams, additives always play a crucial role. They ensure proper reactivity, morphology, stability, thermal resistance and mechanical performance.

Although the primary components of polyurethane (polyol and isocyanate), determine the chemical backbone, the final properties are heavily conditioned by the additive package, even if it represents a small amount of the total system.

Most relevant classes of additives in spray polyurethane systems include: surfactants and silicone stabilizers, catalysts, flame retardants and blowing agents.

3.2.3.1.Surfactants and silicone stabilizers

These compounds are commonly silicone-based surfactants, they provide excellent thermal stability to polyurethane foams, stabilizing the growing foam’s cell structure during the expansion phase.

Its functions include the control of cell size and cell uniformity, the prevention of foam collapse or shrinkage and the maintenance of the balance between open and closed cells. This last point is particularly critical for thermal insulation properties, therefore for SPF.

The stabilizing effect arises from the silicone-alkylene oxide structure, where the silicone backbone is hydrophobic and the polyether side chains are hydrophilic. This amphiphilic nature allows the molecules to orient at gas-liquid interfaces, which reduce surface tension and stabilizes bubble formation.

In SPF systems, where the reaction speed is very high and environmental conditions can be variable (for example temperature and humidity), stabilizers are essential to guarantee cell consistency and mechanical properties across the surface of the foam.

3.2.3.2. Catalysts

Catalysts accelerate the whole process of polyurethane foam formation. The balance between the urethane formation and urea and CO_2, often referred as gelling and blowing respectively, is critical in spray applications.

Amine-based catalysts (e.g., triethylenediamine, dimethylcyclohexylamine), promote both gelling and blowing reactions. Some amines are more selective for the water-isocyanate reaction. Metal-based catalysts (e.g., dibutyltin dilaurate, potassium octoate), mainly catalyse the isocyanate-polyol reaction. They are effective but less commonly used in spray systems, as it response is slower under ambient conditions.

The reaction profile in spray systems must be carefully optimized: too fast reactions may cause poor mixing and uneven expansion, on the other hand, too slow reactions may result in foam sagging and poor adhesion to the substrate.

For this reason, usually blends of amine catalysts are typically tailored for SPF systems, ensuring an equilibrium between a rapid rise time, a good surface cure and a controlled foam growth.

3.2.3.3. Flame retardants

Polyurethane foams are inherently flammable due to their high organic content, therefore flame retardants are mandatory in order to enhance fire performance, by reducing the heat release rate and limiting flame propagation.

Several possibilities are available for flame retardancy:

- Aromatic polyester polyols: Aromatic rings within the backbone of the polyester polyols promote char formation during thermal decomposition. When the material is exposed to heat or flame these structures resist decomposition better than aliphatic ones. Additionally, upon degradation they tend to form a stable carbonaceous char layer, which acts as a protective shield that slows heat transfer to the underlying foam and reduces the release of volatile and flammable gases.

- Phosphorous-based compounds: These compounds decompose in several species during combustion, forming phosphoric acid derivatives and radicals that eventually inhibit flame propagation in the gas phase. This interrupts the radical chain reactions in the flame. These compounds are often described as having synergistic effects when used in combination with aromatic polyols. A widely used compound is TCPP (Tris(1-chloro-2-propyl)phosphate.
- Halogenated flame retardants: Release halogen radicals during combustion, again, interrupting the flame radical chain reactions.
- Intumescent agents: These compounds make the foam expand under heat, forming a char barrier that protects the underlying foam. It is highly effective in stopping the flame to propagate.

3.2.3.4. Blowing agents

Blowing agents are essential components in many SPF systems. They are responsible for generating the gas phase that creates the foam's cell structure. Its impact is very significative on foam density, dimensional stability, thermal conductivity and also environmental compliance.

They can be divided into two classes: chemical agents (the more important being water, forming CO_2) and physical agents. This last class has evolved through several technological generations, primarily driven by environmental regulations and the need for higher performance insulation.

Physical agents consist of a liquid or a gas with a very low boiling point, that evaporates by the released heat of the reactions that take place in polyurethane formation. This gas remains ideally trapped into the cells of the foam. However, with time they are released, and some of them are responsible for accelerating the greenhouse effect.

As mentioned before this class has evolved over time, and we can distinguish four generations:

- First generation: Chlorofluorocarbons (CFCs), provided excellent insulation properties due to their low thermal conductivity. However, they were ozone-depleting substances with high global warming potential, so they are currently banned.
- Second generation: Hydrochlorofluorocarbons (HCFCs), although being less responsible for ozone depletion, they were not environmentally benign. Again, by the early 2000s they were almost out of the market.
- Third generation: Hydrofluorocarbons (HFCs), not considered to be responsible for ozone depletion. Their chemical stability, compatibility with polyol systems and

favourable processing characteristics made them particularly suitable for its use in polyurethane foams. However, they are considered to have a high global warming potential.

- Fourth generation: Hydrofluoroolefins (HFOs), are more environmentally friendly alternative. They have zero ozone depletion potential and lower global warming potential. HFOs are now the preferred physical blowing agents, particularly in residential, commercial and industrial insulation applications that require compliance with green building standards.

Other options include compounds that do not belong to any conventional generation of blowing agents. Pentane-based agents offer a valuable balance between thermal conductivity performance and regulatory compliance, particularly due to their low global warming potential (GWP) and zero ozone depletion potential (ODP). The physical properties of pentane also contribute to stable foam expansion and a uniform cell structure.

However, the main drawback is its flammability, which requires manufacturers to operate in specialized and properly equipped facilities in order to handle it safely as a blowing agent.

Co-blowing strategies are also employed by combining physical blowing agents like pentane with water or with HFOs/HFCs. This dual approach enables control over foam density, reactivity, and closed-cell content. Additionally, it can optimize processing conditions (for example, improving foam adhesion to the substrate under low-temperature conditions) while helping to reduce raw material costs without significantly compromising performance.

4. Experimental methods and characterization techniques

This section describes the experimental procedures and analytical methods applied to evaluate the properties and performance of the SPF systems developed in this project. Each one is performed in order to provide critical insight into the behaviour, processability, safety, and final performance of the resulting sprayed foams. Additionally, several of these tests are required to ensure compliance with relevant technical standards and regulatory frameworks, particularly those concerning thermal insulation, fire performance and dimensional stability. These aspects will be discussed in greater detail in the following section.

4.1.Moisture content

Moisture in the polyol blend (that is, polyol and additives), as it has been discussed in the previous section, gives rise to specific reactions that affect density and other aspects of the final product. Therefore, it is a crucial parameter that has to be measured and adjusted.

In spray polyurethane systems, typical moisture content is generally between 1.5% and 3.5%, depending on the formulation.

The Karl Fischer titration method was used, which allows precise quantification of trace amounts of water in polyols, following the procedure described in ISO 14897:2003. [7]

4.2.Viscosity

Dynamic viscosity it is determined using a rotational viscometer at a constant temperature of 25ºC, since this property is strongly temperature-dependent. This parameter reflects the flow behaviour and mixing efficiency of the polyol blend with the isocyanate during the spraying process. High viscosity values are avoided, because they may hinder homogeneous mixing and can lead to poor foam formation along with several machine-related problems. For SPF systems, typical values fall within the range of 150 and 600 mPa·s.

Viscosity of the polyol was determined using an Anton Paar Rheolab QC rotational rheometer, according to the procedure specified in ISO 3219:2013. [8]

4.3.Specific density

Specific density of the polyol mixture is measured in a densimeter. This value is crucial for conducting the experiments, because polyurethane spray machines operate with a 1:1 volume ratio. Knowing the densities of the polyol and the isocyanate, calculations can be made in order to use the same volume for both compounds.

Typical values range between 1.1 and 1.3 g/cm^3.

In this case, a Mettler Toledo DA-100M density meter was used, following the procedure described in DIN 51757. [9]

4.4.Flash Point

The flash point of a polyol blend is assessed following the EN ISO 3679 [10], using the closed-cup method in equilibrium. The apparatus (Setaflash Series 3) heats the sample within a sealed vessel, mimicking the conditions of a closed container. This test identifies the lowest temperature at which the vapour above the sample ignites in the presence of an ignition source.

The flash point provides critical safety information for handling, storage, and transport of the liquid formulation. It is a regulatory requirement for classification and labelling.

A wide range of values could be obtained depending on the components of the polyol system. If the value does not exceed 60ºC, the polyol system has to be classified as flammable.

4.5. Free spray foaming: reactivity and density assessment

A free spray foaming test is performed in order to evaluate the reactivity, visual aspect, and density of each experimental polyurethane formulation. In the case of our laboratory, it is done in a plastic cup of 320 cm^3.

Reactivity evaluation consists of recording three characteristic times:

- Cream time (start time): The time elapsed from the moment the polyol and isocyanate components are mixed until the first visible signs of foam expansion or a noticeable change in colour appear.
- Gel time: The time at which the reacting foam mixture transitions from liquid to solid-like behaviour. This is evaluated by repeatedly inserting and withdrawing a thin metallic stick into the foam. When the stick is removed and thin filaments of material form, gel time is registered.
- Tack-free time (cure time): The point at which the foam surface is no longer sticky to the touch, this indicates the onset of structural stability.

Visual inspection is qualitatively evaluated by searching for uniformity of cell structure, presence of surface defects or any shrinkage, and colour consistency (last one to ensure proper mixing).

Finally, free-rise density measurement takes place after the foam has fully cured. Curing implies the reaction has finished and the skin of the polymer is solid.

Figure 6. Example of free-rise spray foams

4.6. Small burner fire test

The fire behaviour of the foam it is evaluated using the small burner test, following the procedure described in the EN ISO 11925-2 [11], and taking into account the classification criteria established in UNE EN 13501-1. [12]

This process assesses the ignitability of building materials exposed to a small flame. The test consists of applying a direct flame to the surface or edge (in the case of SPF systems, only the surface) of the specimen for 30 seconds, and measuring the flame spread and self-extinguishing behaviour. To achieve Euroclass E classification, the flame propagation must not exceed 150mm within 60 seconds. Observations on smoke generation and material degradation are also recorded.

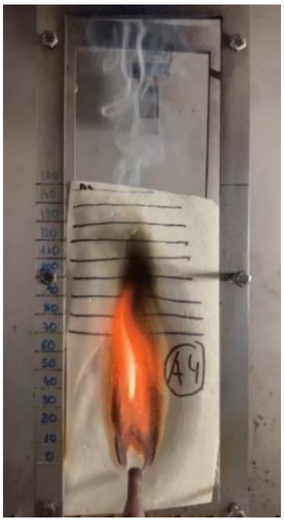


Figure 7. Small burner fire test

It is prepared from a free-rise foam poured into a paper box, from which the test samples are cut without removing the foam’s skin.

For safety reasons, all measurements are conducted inside a closed metalic cabinet equipped with an extraction system. This system is activated after 60 seconds to ensure adequate ventilation and prevent the accumulation of potentially harmful vapors.

4.7.Spraying of test panels and subsequent characterization

Square polyurethane foam panels are sprayed using the experimental formulations. These panels serve as standardized specimens for a series of mechanical and thermal tests. The application process is carried out at controlled ambient conditions and consistent substrate temperatures to ensure reproducibility.

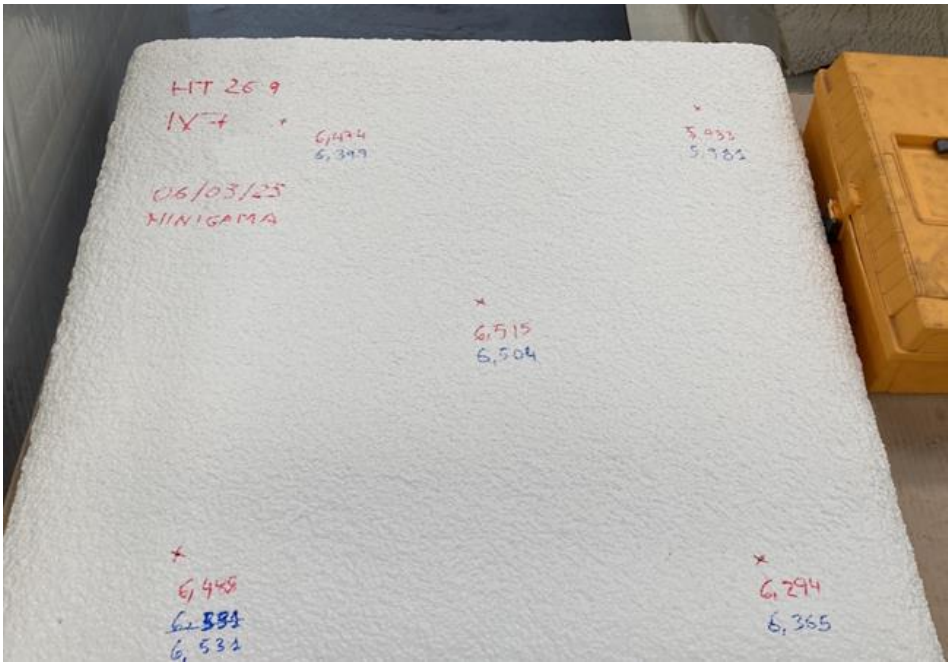


Figure 8. Example of a panel of an SPF system

The following is a description of several tests carried out on the spray polyurethane foam panels to evaluate their performance.

4.7.1. Density

The density is measured by cutting representative approximately cubic samples from the foam panel and calculating the ratio between their mass and volume, usually by Arquimides' principle, as the density is determined from specimens that include the full thickness of the panel and have an irregular upper face.

This test follows the procedure specified in the UNE 921920-08. [13]

4.7.2. Thermal conductivity

The thermal performance is assessed by determining the thermal conductivity (λ-value) of a sample of specific dimensions: 200 x 200 x 30 mm, using UNE-EN 12667. [14]

The sample is previously conditioned under controlled temperature and moisture for a minimum of 48 hours and a maximum of 8 days to ensure thermal equilibrium and moisture stability. Then it is placed in an apparatus intended for this application, a Netzsch HFM Lambda. The sample lies between two plates (hot and cold), and the heat flux is measured via sensors in contact with the specimen surfaces.

By using Fourier's law, thermal conductivity is calculated according to Equation 1:

$$\lambda = \frac{Qd}{A\Delta \mathrm{T}} \tag{1}$$

Where:

- λ = thermal conductivity (W/m·K)
- Q = heat flow rate (W)
- d = thickness of the specimen (m)
- A = surface area through which heat flows (m^2)
- ΔT = temperature difference across the sample (K)

The final value is reported and expressed at 10ºC mean temperature (between the two plates), as λ_{10}, in compliance with EU regulations for thermal insulation materials.

This measurement provides a critical parameter for evaluating the insulating performance of the SPF system. Lower λ-value indicate better thermal resistance. Typical values lie within the range of 0.0190 to 0.0220 W/m·K.

4.7.3. Normality test

The UNE-EN 14315-1 [15] stablishes alternative thermal ageing procedures. One is the accelerated ageing procedure, that consists of measuring the initial λ and then age the sample 175 days at 70ºC. But there is another option, which is often preferred.

The fixed increment procedure stablishes that, after measuring the initial λ, Normality test can be performed, and consists on 21 days ageing at 70ºC on a 20mm core foam. If the increase in λ is below stablished limits then a table that includes several values depending on the used blowing agent and the cut of the foam sample is used to add a fixed increment to the initial value.

Normality test is designed for products blown with blowing agents that are considered "permanent" (e.g. HFO and hydrocarbons such as pentanes).

4.7.4. Dimensional stability

The dimensional stability of the spray-applied polyurethane foam is evaluated following the EN 1604 [16]. This test assesses the material's ability to retain its shape and size when subjected to thermal stress.

Two samples were measured (with a digital calliper) and subsequently exposed to different conditions: the first was placed in a laboratory oven at 70 °C, and the second in a conventional freezer at −20 °C, both for a duration of 48 hours. After exposure, the samples were measured again to evaluate potential dimensional or physical changes. The percentage change in dimensions has to be calculated, and then compared against the maximum allowable dimensional changes defined in EN 14315-1.

4.7.5. Compressive strength

The compressive strength of the spray-applied polyurethane foam is evaluated in accordance with EN 826 [17], which specifies the procedure for determining the compressive behaviour of thermal insulation products. It provides an insight into the material's ability to withstand mechanical loads and maintain dimensional integrity in structural or pressure-bearing applications.

Mechanical tests were carried out using a Tinius Olsen H25KS universal testing machine.

Samples of 50 x 50 x 50 mm are cut from the cured foam panel, such that the compressive force is applied perpendicular to the surface exposed during spraying. A uniaxial load is applied at a controlled rate until the specimen reaches 10% of deformation. The compressive stress at 10% strain is recorded and reported in kPa. This is especially important to evaluate situations where they may be mechanical constraints, such as floor insulation. It also provides information about cell structure integrity, which influences compressive performance.

Table 1. Classification levels depending on compressive strength

Level	Requirement kPa
CS (10/Y)100	≥ 100
CS (10/Y)150	≥ 150
CS (10/Y)200	≥ 200
CS (10/Y)300	≥ 300
CS (10/Y)400	≥ 400
CS (10/Y)500	≥ 500

4.7.6. Open cell content

The open cell content of the polyurethane foam is determined following the methodology described in ISO 4590 [18]

The test involves preparing samples of known geometric volume and measuring the air-impermeable volume using a gas displacement apparatus (which uses nitrogen). The difference between the two values allows for the calculation of the volume percentage of open cells and closed cells.

Figure 9. High open cell content on a flexible foam

Depending on these percentages, products can be classified as shown in Table 1:

Table 2. Classification classes depending on closed-cell content

Class	Closed cell content
CC1	< 20%
CC2	20% to 80%
CC3	> 80% to 89%
CC4	≥ 90%

Each classification indicates the expected thermal insulation performance and moisture resistance of the foam, being the CC4 class the one with better performance.

4.7.7. Small fire burner test

The same procedure described in section 3.6 is used. This time, the specimens are cut directly from the sprayed foam panel.

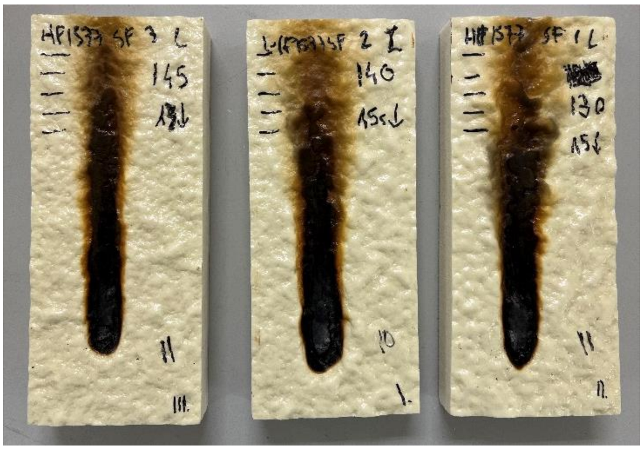


Figure 10. Example of three samples subjected to a flame behaviour test

5. Regulatory Framework and Technical Documentation

The development, commercialization, and legal distribution of spray polyurethane foam (SPF) systems within the European Union require compliance with standardized technical and regulatory frameworks. These frameworks are established to ensure product safety, environmental responsibility, and performance consistency across construction projects.

Among these, the EN 14315-1:2013 standard, which specifies the requirements for in-situ applied thermal insulation products made of polyurethane (PUR) and polyisocyanurate (PIR), stands as the central normative reference for CE marking within the European Union.

This section aims to provide a comprehensive analysis of the mentioned standard, summarizing its structure, objectives, and key technical parameters, and explaining the documentation required to demonstrate conformity, including the Declaration of Performance (DoP) and the Technical Datasheet (TDS). The alignment of the formulations developed in this project with the regulatory requirements is a fundamental condition for their potential market application and industrial scalability.

5.1. EN 14315-1:2013 – Scope and Technical Criteria

EN 14315-1:2013 is a harmonized product standard under Regulation (EU) No 305/2011 (the Construction Products Regulation, CPR), which governs thermal insulation products manufactured in situ by spray application of polyurethane (PUR) or polyisocyanurate (PIR) foam. This standard sets out the essential characteristics such products must meet in order to obtain CE marking and ensure their lawful use across European markets.

The standard applies to spray-applied rigid foam systems used primarily for thermal insulation of building envelopes, including roofs, walls, floors, and ceilings. It defines the methods for testing, assessing, and declaring performance characteristics such as thermal conductivity, compressive strength, closed-cell content, dimensional stability, and fire reaction classification. These criteria form the basis for regulatory compliance and market access.

5.1.1. Scope of Application

The standard covers polyurethane and polyisocyanurate insulation products formed by spraying directly onto the construction surface. Its applicability spans a range of applications in both new builds and renovation projects, particularly where complex geometries or high-performance insulation is required.

Common applications include:

- Thermal insulation of pitched and flat roofs
- Insulation of internal and external vertical surfaces
- Underfloor insulation
- Ceilings, crawl spaces, attics, and basement walls
- Passive house retrofits and airtightness enhancement

5.1.2. Classification System

EN 14315-1 provides a detailed classification scheme that enables the objective assessment of product performance. These classes are based on quantifiable parameters, each with prescribed test methodologies and declaration formats. They serve as benchmarks for design professionals and regulatory bodies to evaluate compliance and applicability in specific construction environments.

The key classification parameters include:

- Thermal conductivity (λ-value)
- Compressive strength
- Closed-cell content
- Reaction to fire
- Dimensional stability
- Water absorption
- Water vapor diffusion resistance factor (μ)

A comprehensive classification enables specifiers to match the insulation product with the performance needs of each construction element.

5.2. CE Marking Requirements

The CE mark is a legal requirement for construction products sold within the European Economic Area (EEA) and indicates conformity with EU regulations. For products under EN 14315-1, the path to CE marking follows System 1 of Assessment and Verification of Constancy of Performance (AVCP), the most rigorous of the AVCP systems. This includes both third-party certification and ongoing factory inspections.

To obtain the CE mark, the following steps are mandatory:

- Initial Type Testing (ITT): This involves laboratory testing of a representative sample of the product to determine its declared performance characteristics. Testing must follow the procedures outlined in EN 14315-1 and relevant horizontal test standards.
- Factory Production Control (FPC): The manufacturer must implement and maintain an FPC system to ensure consistent production quality. The FPC includes equipment calibration, operator training, batch traceability, and routine internal testing.
- Notified Body involvement: A Notified Body must audit the production process, verify the ITT results, and issue a certificate of constancy of performance. Regular surveillance audits follow.
- Declaration of Performance (DoP): The manufacturer drafts a legal document declaring the product’s performance values, which must be publicly accessible.
- Affixing the CE marking: The CE mark and relevant performance classes must be shown on packaging, accompanying documentation, or directly on the product if

possible. The product label must include at least the CE mark, reference to EN 14315-1, the declared thermal performance, and the identification number of the Notified Body involved.
- Failure to comply with these steps can lead to product recalls, fines, or bans on marketing and sale.

5.3. Declaration of performance (DOP)

The Declaration of Performance (DOP) is the cornerstone document that links a construction product to its declared performance and CE mark. It is required under the CPR and must be available for all CE-marked products, in print or digital form. The DoP serves as a guarantee to designers, builders, and regulatory authorities that the insulation system performs as claimed.

The DoP must include:

- Product identification code
- Intended use as specified in the harmonized standard
- Manufacturer’s name and address
- System of assessment and verification (System 1 for EN 14315-1)
- Declared performance values for each essential characteristic
- Reference to the Notified Body and certification details
- Signature of the legally authorized representative

5.4. Technical Data Sheet (TDS)

The Technical Data Sheet (TDS), in industrial and construction settings, is key for comprehensive technical and application-specific information to architects, installers, and technical assessors. It also provides health and safety guidance and operational recommendations, especially for reactive chemical systems like polyurethane foams.

A typical TDS for a spray polyurethane system includes:

- System description and formulation type (e.g., 2-component, HFO-blown, aromatic)
- Mixing ratio by volume or weight
- Processing parameters: temperature ranges, substrate preparation, environmental conditions (according to EN 14315-2)

- Reaction profile: start time, gel time, tack-free time, foam rise
- Declared properties: λ-value, CCC level, density, compressive strength, adhesion to substrate, fire classification
- Compatibility with substrates and coating systems
- Storage, shelf life, and packaging instructions
- Health and safety notes, including handling of isocyanates and reference to the SDS

The TDS must be updated regularly and must indicate the DoP to which it refers.

5.5. Integration with the Project Objectives

The integration of regulatory compliance within the research and development process is a central aim of this project. By referencing EN 14315-1 throughout the experimental formulation work, the project ensures that all tested systems align not only with theoretical expectations but also with real-world commercial and legal requirements.

Moreover, understanding the CE marking pathway provides insights into future industrial transfer scenarios.

Ultimately, regulatory alignment it is not treated as an administrative requirement, but as a core design parameter—ensuring that every formulation developed has the potential to reach the market swiftly, legally, and competitively.

6. Results and discussion of the experimental studies

This section presents a description and the outcomes of the experimental studies carried out on the various SPF formulations. To ensure confidentiality, the commercial names of both the final products and the raw materials have been modified.

The first and second subsections are dedicated to clarifying the nomenclature used throughout the study. Third subsection provides a detailed description of each experimental study, including the corresponding results. A fourth subsection offers a discussion of their implications in terms of performance, formulation optimization, and compliance with application requirements.

6.1. Nomenclature of raw materials

To preserve confidentiality, all raw materials used in the experimental studies have been assigned simplified, code-based identifiers. These codes indicate the chemical nature or functional role of each component within the formulation, following a consistent logic:

- PE-A# refers to aminic polyether polyols, where the number distinguishes different variants; FR indicates flame retardancy.
- PES-# corresponds to polyester polyols, with numbering used to differentiate between aromatic and aliphatic types, as well as viscosity grades.
- ADD-# identifies additives, such as flame retardants (FR) or crosslinkers.
- SIL-# denotes silicone-based stabilizers.
- HFO stands for the main fourth-generation blowing agent used.
- C-BA is used for the co-blowing agent, selected for its cost-efficiency.

Table 3. Assigned codes and characteristics of the raw materials used in the formulations

Assigned code	**Characteristics**
PE-A1	Aminic aromatic polyether
PE-A2-FR	Aromatic, aminic (Mannich) polyether, flame retardant
PE-A3	Aminic aromatic polyether
PE-A4	Aminic polyether, low viscosity
PES-1	Aromatic polyester, high viscosity
PES-2	Aromatic polyester, low viscosity
PES-3	Aliphatic polyester, low viscosity
ADD-1	Crosslinker
SIL-1	Silicone-based stabilizer
SIL-2	Silicone-based stabilizer
SIL-3	Silicone-based stabilizer
SIL-4	Silicone-based stabilizer
HFO	4th generation blowing agent (HFO), high price
C-BA	Co-blowing agent, low price

6.2. Nomenclature of SPF systems

The spray polyurethane foam (SPF) product portfolio considered in this work can be divided into two main families: blowing agent-based (BA) formulations and water-based (W) formulations. Within the BA family, BA-WE1 serves as the reference formulation for wall and enclosure applications (WE). On the other side, BA-WE2, is a cost-optimized formulation that maintains the same application scope but achieves a cost reduction when combined with the co-blowing agent evaluated in this project.

From BA-WE1, a derivative was developed, BA-RF1, for roofing and flooring applications (RF), with modified properties to meet different application demands. In turn, BA-RF2 was developed from BA-WE2 as a lower-cost alternative to BA-RF1, targeting the same application areas. In the water-based family, W-M is a product with a more versatile profile, suitable for both wall and enclosure as well as roof and terrace applications (M stands for mixed).

The experimental work presented in this study focuses on four of these formulations: BA-WE2, BA-RF1, BA-RF2, and W-M.

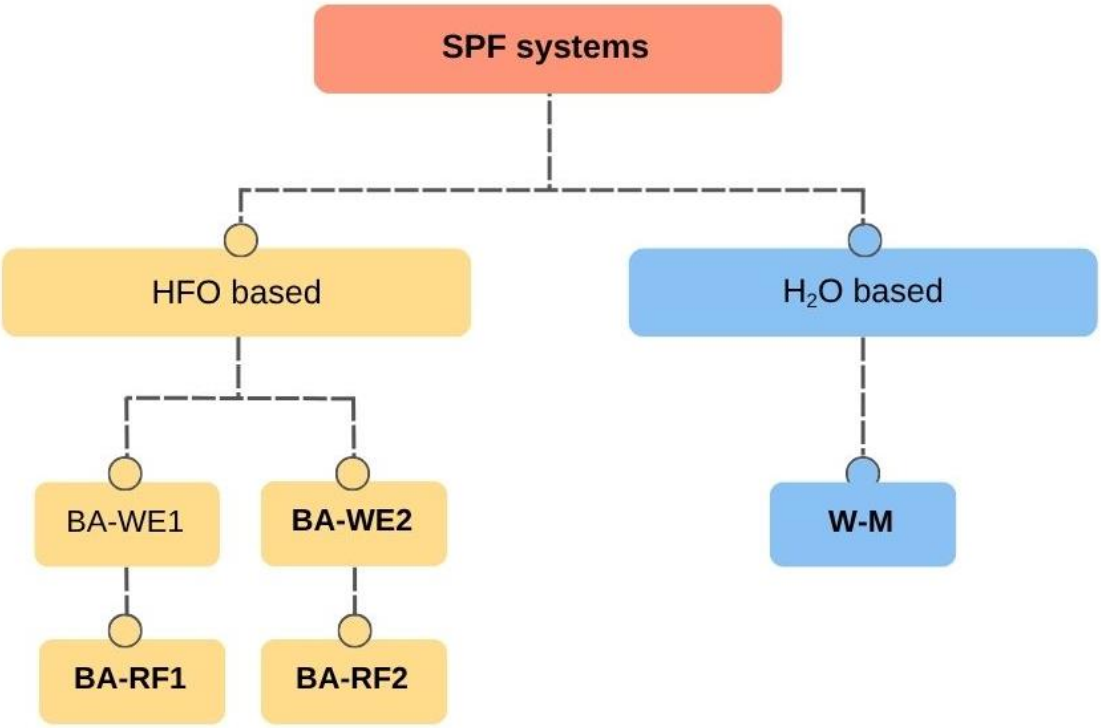


Figure 11. Spray polyurethane foam systems scheme

As was done with the raw materials, all SPF formulations evaluated in this study have been assigned simplified, code-based identifiers to preserve confidentiality. In this case, the codes refer to the final spray products developed and tested, and are structured to reflect both the type of blowing agent used and the main application field.

The naming logic is as follows:

- BA-WE# refers to blowing agent-based formulations intended for wall and enclosure applications.
- BA-RF# designates blowing agent-based systems designed for roofing and flooring.
- W-M denotes water-based multi-purpose formulations, applicable across walls, roofs, and terraces.

Table 4. Assigned codes and characteristics of SPF systems

Assigned code	Characteristics
BA-WE1	Base blowing agent-based formulation for wall and enclosure applications
BA-WE2	Cost-reduced alternative to BA-WE1 for wall and enclosure applications
BA-RF1	Derivative of BA-WE1 adapted for roofing and flooring applications
BA-RF2	Cost-reduced alternative to BA-RF1, derived from BA-WE2
W-M	Water-based formulation, suitable for walls, roofs, and terraces

6.3. Experimental studies

The experimental studies conducted are divided into two categories: manual and physical. In the laboratory, these are commonly referred to as HM and HF, respectively. Both abbreviations derived from the German word *hart* ("hard"), with HM corresponding to manual studies and HF to physical studies.

HM studies involve preparing an SPF formulation from raw materials in the laboratory. During these tests, the reactivity profile and foam density are evaluated and adjusted if necessary. Fire behaviour is also commonly assessed at this stage. It is also possible to include in these studies some compressive strength test.

HF studies, on the other hand, focus on the evaluation of the projected foam panels, including tests such as compression strength, open-cell content, and other relevant physical properties.

6.3.1. HM-1

In the HM-1 experimental study, the system BA-RF2 was developed as a derivation of BA-WE2, with the aim of adapting the original wall and enclosure formulation to new performance requirements for roofing and flooring applications. Several compositional modifications were introduced to enhance mechanical strength, fire resistance, and dimensional stability under load.

The primary goal of the test was to identify an alternative to the BA-RF1 product that enables more competitive pricing in the market.

A significant reduction in the content of both blowing agents was implemented: HFO was reduced from 8.5 % to 5.0 % and water from 2.7 % to 1.6 %. This reduction plays a critical role in increasing the foam's final density, which directly enhances compressive strength—a key requirement in roofing and flooring applications. On the other hand, it also affects to the rest of the formula, because spray polyurethane foam machines operate with a fixed 1:1 volume ratio between the isocyanate and polyol components. Therefore, if the water content in the polyol formulation is reduced, the hydroxyl (-OH) index must be increased to maintain the required stoichiometric balance with the isocyanate's (-NCO) groups under this fixed volume ratio.

The percentage of PE-A1, a high-functionality aromatic polyether polyol, was increased from 9.9 % to 15.5 %, reinforcing the crosslink density of the polymer network and improving its compressive performance. Conversely, PES-1, a polyester polyol, was reduced from 36.5 % to 31.1 %. The amount of PE-A2-FR, a flame-retardant-modified polyol, was increased from 3.5 % to 6.1 % to improve the system's fire resistance.

ADD-2 was also added, a cross-linker that highly contributes to rigidity.

Table 5 shows the main changes in the quantities of the components that had to be adjusted to ensure that the appropriate isocyanate index for effective urethane formation was maintained.

Table 5. Components comparison between systems BA-WE2 and BA-RF2

Component (%)	**BA-WE2**	**BA-RF2**
PE-A1	9.9	15.5
PES-1	36.5	31.1
PE-A2-FR	3.5	6.1
ADD-1	0.0	1.0
HFO	8.5	5.0
Water	2.7	1.6
Remaining components	39.0	39.7

Once the BA-RF2 polyol system was prepared in the laboratory, its reactivity and free-rise density were compared to those of the commercial product BA-RF1. The results, shown in **Table 6**, revealed very similar performance profiles between both systems:

Table 6. Reactivity and density of BA-RF2 and BA-RF1 systems

	BA-RF2	**BA-RF1**
Cream time (s)	2.3	2.5
String time (s)	6.4	5.8
Tack-free time (s)	7.4	7.3
Free-rise density (kg/m³)	55.8	55.5

Although the formulation details of BA-RF1 remain undisclosed, a key point is that this system contains 24.2 % of PE-A2-FR, a flame-retardant polyol that significantly increases production costs. In contrast, BA-RF2 achieves comparable reactivity and density values while using only 6.1 % of the same additive.

The following table presents a comparative summary of the average compression test results between the two formulations: BA-RF2 and BA-RF1. The measurements were performed on the nucleus of the manual free-rise foams (performed in a plastic cup).

Table 7. Comparison between results obtained in compression strength test

	BA-RF2	**BA-RF1**
F10% (N)	315	375
d10% (MPa)	0.263	0.314
Fm (N)	343	393
dm (MPa)	0.281	0.323
Compressive modulus (MPa)	8.92	12.70

In terms of compressive performance, although the results obtained for BA-RF2 not reach the same values as those of the BA-RF1 system, they are nevertheless adequate for the intended application. The compressive strength, stress at 10 % deformation, and modulus values of BA-RF2 remain within a functional range that justifies its use as a lower-cost alternative.

An important point to highlight is that the new formulation BA-RF2 was also evaluated with the small burner test. The result obtained was a burn length of 126 mm, which is considered very satisfactory, as it remains well below the 150 mm threshold required for achieving Euroclass E according to EN 13501-1. The outcome confirms that the BA-RF2 system, despite its cost-optimized composition, maintains acceptable fire performance for general construction use.

Thanks to this optimization, it is estimated that the cost of the BA-RF2 polyol blend results in a savings of approximately €0.50 per kilogram when compared to BA-RF1.

6.3.2. HM-2

The main goal of this experiment is to use a co-blowing agent (C-BA) in the BA-WE2 system, in order to increase cost-efficiency.

Several measurements were conducted to determine the flash point of the BA-WE2 system after the addition of the C-BA additive. Preliminary evaluations had already been carried out to understand how the system behaved under this modification.

Originally, the BA-WE2 formulation included 8.5 % of HFO as the physical blowing agent. Following several experimental trials, it was decided to reduce the HFO content to 7.4 % and incorporate 0.5 % of C-BA. This adjustment was driven by a critical objective: in order for a product to be classified as non-flammable, its flash point must exceed 60 °C.

Table 8. Formulations with different proportions of blowing agents

	Formulations				
Component (%)	**1**	**2**	**3**	**4**	**5**
HFO	8.5	6.4	7.0	7.4	7.9
C-BA	0.0	1.0	0.7	0.5	0.3
Remaining components	91.5	92.6	92.3	92.1	91.8
Flash Point (ºC)	>68	32	36	43	60

If the flash point is between 35 °C and 60 °C, the product may still be declared as non-flammable provided that it passes the sustained combustibility test for liquids. This test assesses whether the product continues to burn after ignition is removed. It is based on standard ISO 9038:2025. [19]

As shown in **Table 8**, a combination of 6.4 parts of HFO and 1.0 parts of C-BA yielded a flash point of 32ºC, and a combination of 7 parts of HFO and 0.7 parts of C-BA yielded a flash point of 36 °C, which, although technically acceptable if the sustained combustibility test is passed, leaves limited safety margin. Therefore, the final optimized formulation—with 7.4 % HFO and 0.5 % C-BA—was selected to balance performance, cost, and regulatory compliance with greater thermal safety.

The sustained combustibility test was performed at our company’s facility in Denmark, obtaining a positive feedback, the modified formulation was officially approved for use. This adjustment has resulted in a notable cost reduction. As a consequence, the new version of the product—featuring 7.4 % HFO and 0.5 % C-BA—has been in active production over the past several months, confirming its technical viability and commercial efficiency.

6.3.3. HM-3

The HM-3 experimental series was broader in scope and can be divided into four distinct stages. Before describing the tests in detail, it is important to clarify some preliminary developments that frame the context of this study.

The use of a co-blowing agent in a water-based system is intended to improve thermal insulation and adhesion of the foam to the substrate at low temperatures. In the case of rigid spray polyurethane foam systems based on water as the blowing agent, a minimum substrate temperature of approximately 15 °C is required to ensure proper foam expansion and adhesion, due to the greater formation of polyurea. With a co-blowing agent, presence of these compounds are reduced, and an effective spray application can be achieved at substrate temperatures as low as 5 °C, improving versatility under cold-weather conditions and broadening the system's applicability.

Following this, a modified W-M system was created. This was done mirroring the methodology used in HM-2, but adapting it to a system without HFO. The new developed formulation will be referred as W-M-LW (that stands for low water).

Another previous experiment was performed in order to develop and evaluate a new cost-optimized variant. This modified formulation will be referred to here as W-M-X. The aim of that initial adjustment was to assess the feasibility of lowering raw material costs without compromising performance, particularly in terms of reactivity and foam density, and improving processability.

6.3.3.1. Part A: Assessment of variations related to W-M formulation

The first phase of the HM-3 experimental study focused on evaluating the reactivity and free-rise density of four different systems:

1. W-M-LW (reference system with low water content, A1)
2. W-M-LW + C-BA (reference with co-blowing agent, A2)
3. W-M-X (cost-optimized variant, A3)
4. W-M-X + C-BA (cost-optimized with co-blowing agent, A4)

It is important to note that the systems containing C-BA—a flammable additive—were not produced at our local facility due to handling restrictions at the time. Instead, they were formulated and prepared at our Italian branch, and then transported for testing.

All four systems were sprayed prior to the test under identical conditions to assess their behaviour.

Obtaining similar measurements of reactivity and density, it was decided to subject the variants of modified formula (W-M-X and W-M-X + C-BA) to fire testing, in order to determine their compliance with the 150 mm limit for Euroclass E classification under EN 13501-1.

There are two standard orientations (two test types) for burning foam samples during fire testing: edgewise and surface (superficial) exposure to the flame. However, in spray polyurethane foam systems, only the surface burning configuration is used, as it better represents real application conditions.

Table 9. Results of small burner fire test of W-M-X and W-M-X + C-BA systems

	W-M-X (A3)			**W-M-X + C-BA (A4)**		
Samples	3			3		
Test type	Superficial			Superficial		
Flame spread (mm)	145	140	145	>150	>150	>150
Surf. of damage (mm)	110	110	115	110	122	122
Flame duration (s)	16	15	15	14	15	16
Classification	E			Not E		

Unfortunately, the version containing the co-blowing agent did not pass the test, as the flame spread exceeded the 150 mm threshold, disqualifying it from being declared as non-flammable according to current CE marking requirements.

6.3.3.2. Part B: Fire performance improvement trials on the W-M system

In this phase of the study, several trials were carried out in an attempt to improve the fire performance of the W-M system by modifying the surfactant package. Two different silicone-based surfactants were tested: SIL-1, the standard surfactant used in the formulation, and SIL-2, an alternative option.

Table 10. W-M system variations with different silicones

	Formulation			
Component (%)	**B1 (std)**	**B2**	**B3**	**B4**
SIL-1	1.0	0.5	0.0	0.0
SIL-2	0.0	0.5	1.0	1.0
C-BA	0.0	0.0	0.0	2.0
Remaining components	99.0	99.0	99.0	98.0

The experimental plan included replacing SIL-1 with SIL-2, as well as testing combinations of both surfactants. Additionally, SIL-2 was tested in the W-M + C-BA formulation, which contains 2 parts of the co-blowing agent C-BA.

Table 11. Results of small burner fire test of variations relying on different silicones

	Formulation											
	B1			**B2**			**B3**			**B4**		
Samples	3			3			3			3		
Test type	Surface			Surface			Surface			Surface		
Flame spread (mm)	120	130	130	120	125	125	135	120	115	140	135	140
Surf. of damage (mm)	120	110	90	100	100	110	80	90	120	100	110	110
Flame duration (s)	19	20	20	19	19	20	20	20	19	12	14	10
Classification	E			E			E			E		

Despite these variations, no significant improvement in fire behaviour was observed across the different configurations. As a result, the strategy of enhancing fire performance through surfactant modification has been put on hold for the time being.

6.3.3.3. Part C: Fire performance improvement trials on the W-M-X system

In the third phase of the HM-3 study, the focus returned to the cost-optimized formulation W-M-X. As in the previous stage, a series of tests were conducted using the surfactants SIL-1 and SIL-2, followed by fire performance evaluation.

Before discussing the results, it is important to clarify the changes that define the transition from the original W-M formulation to the new variant W-M-X.

Compared to the W-M formulation, W-M-X includes a slight increase in the content of PE-A3 (from 23.0 % to 25.1 %), while completely removing PES-3 and PE-A4. In their place, 28.8 % of PES-2 is introduced. The following table summarizes these compositional differences:

Table 12. W-M and W-M-X formulations comparison

Component (%)	**W-M**	**W-M-X**
PE-A3	23.0	25.1
PES-3	15.7	0.0
PE-A4	15.0	0.0
PES-2	0.0	28.8
Remaining components	46.3	46.1

Changes introduced in the W-M-X formulation are aimed at significantly reducing raw material costs while attempting to preserve the system’s overall performance. The polyether polyol PE-A3 is a relatively cost-effective component that contributes to structural consistency without substantially increasing the formulation cost. In contrast, both PES-3 and PE-A4 are aminic polyols that are expensive, and their removal contributes directly to cost savings. They also presented another problem: they were used only for this system. As a replacement, PES-2 (an aromatic polyol) is used in higher proportion. It represents a more affordable alternative that can offer comparable mechanical behavior when the formulation is properly adjusted with the appropriate balance of catalysts and surfactants. While these substitutions aim primarily at cost reduction, the formulation was designed to maintain acceptable fire performance.

Proportions of components, including SIL-1, SIL-2, and the rest of the formulation, are the same as those listed in **Table 10**. being in total four different variations of the W-M-X system.

The results of the small burner test are presented in **Table 13**.

Table 13. Results of the small burner fire test of W-M-X variations relying on different silicones

	Formulation											
Parameter	**C1**			**C2**			**C3**			**C4**		
Samples	3			3			3			3		
Test type	Surface			Surface			Surface			Surface		
Flame spread (mm)	115	125	130	135	125	125	120	130	135	150	140	140
Surf. of damage (mm)	70	90	100	130	100	100	90	90	90	120	120	110
Flame duration (s)	14	15	15	15	14	16	15	16	14	33	22	21
Classification	E			E			E			E		

As shown, Formulation 4 (which corresponds to the same composition as Formulation 3 but with the addition of C-BA), yields results that are too close to the regulatory limits. Based on these findings, the use of the standard formulation in combination with a co-blowing agent (W-M-X + C-BA) is currently being considered for markets outside the European Union, where CE marking is not required. At present, further testing with this product is being carried out as part of a separate experimental study.

6.3.3.4. Part D: Continuation of the fire performance improvement trials on the W-M-LW system

In this final stage of the HM-3 experimental study, the investigation continued with a focus on improving the fire behaviour of the W-M-LW system containing C-BA. Building on a similar methodology to that one used in Parts B and C, a new series of formulations were prepared, each containing 2.0 % of C-BA and differing only in the type of silicone surfactant used. The goal was to evaluate whether changing the surfactant could lead to better fire resistance in the presence of the co-blowing agent.

The three formulations tested are summarized in the following table. All formulations maintained a constant percentage of C-BA (2.0 %), while the type of silicone varied among SIL-2, SIL-3, and SIL-4, each used at 1.0 %. The remaining components in the formulation accounted for 97.0 % in all cases.

Table 14. W-M-LW system variations with different silicones

	Formulation		
Component (%)	**D1**	**D2**	**D3**
SIL-2	1.0	0.0	0.0
SIL-3	0.0	1.0	0.0
SIL-4	0.0	0.0	1.0
C-BA	2.0	2.0	2.0
Remaining components	97.0	97.0	97.0

The results of these three formulations on the small burner test are presented in **Table 15**.

Table 15. Results of the small burner fire test of W-M-LW variations relying on different silicones

	Formulation								
	D1			**D2**			**D3**		
Samples	3			3			3		
Test type	Superficial			Superficial			Superficial		
Flame spread (mm)	135	130	140	130	120	130	120	125	130
Surf. of damage (mm)	120	120	110	120	120	110	70	90	90
Flame duration (s)	14	14	14	12	12	14	8	8	8
Classification	E			E			E		

As shown in the results, the formulation containing SIL-4 yielded the best fire performance overall, achieving the lowest average flame spread, smallest extent of surface damage, and shortest flame duration among the variants tested.

Currently, Formulation D3 (which incorporates SIL-4) is undergoing aging evaluation. In this study, a liquid sample of the formulation is stored for three months in an oven at 45 °C, simulating accelerated aging conditions. Throughout this period, the reactivity, free-rise density, and visual appearance of the foam are monitored on a monthly basis, in order to assess the stability of the formulation components when exposed to prolonged high temperatures.

6.3.4. HF-1

The HF-1 study involved a series of physical tests conducted on two sprayed foam panels: one produced using the BA-WE2 formulation, and the other using BA-WE2 + C-BA. The latter formulation was previously defined during the HM-2 experimental study, where the optimal C-BA content was established.

Although the product already holds a valid CE marking, and the addition of a small proportion of C-BA does not require repeating the full initial type testing, the HF-1 tests were conducted in order to support and update the Declaration of Performance (DoP). This ensures that the product's characteristics—including the influence of the C-BA—can be transparently reported in accordance with applicable regulatory obligations.

In this test campaign, the following properties were evaluated for both panels:

- Apparent density
- Dimensional stability
- Thermal conductivity
- Compressive strength
- Small burner fire behaviour
- Open-cell content

These tests provide a comprehensive evaluation of the mechanical, thermal, and structural performance of both systems under standardized conditions.

6.3.4.1. Density

As shown in the table, the measured densities with skin of the two sprayed systems are quite similar, indicating good consistency between the formulations. The recorded values—

51.1 kg/m³ and 48.1 kg/m³—fall within the expected range for this type of application. It can be seen that using a co-blowing agent reduced density about 3 kg/m³.

Table 16. Density results for BA-WE2 and BA-WE2 + C-BA systems

	BA-WE2	BA-WE2 + C-BA
Density with skin (kg/m³)	51.1	48.1
Total thickness (mm)	68	70
Number of layers	7	7

The table also indicates the number of layers required to achieve an approximate total thickness of 70 mm, both systems required the same number to reach the target thickness, confirming similar sprayability. All spray applications were performed in-house at our company's facilities, under controlled conditions to ensure representative results for subsequent physical testing.

6.3.4.2. Dimensional stability

In dimensional stability tests, both systems presented acceptable volume variation under thermal stress.

Table 17. Dimensional stability results for BA-WE2 and BA-WE2 + C-BA systems

	BA-WE2	BA-WE2	BA-WE2 + C-BA	BA-WE2 + C-BA
Temperature (ºC)	70	-20	70	-20
Time (h)	48	48	48	48
Change in V (%)	-7.32	-0.19	-9.17	-0.54

6.3.4.3. Thermal conductivity

The thermal conductivity results indicate that both systems deliver consistent insulating properties, with very slight differences in λ. These differences suggest equivalence in thermal insulation capacity.

Table 18. Thermal conductivity results for BA-WE2 and BA-WE2 + C-BA systems

	BA-WE2	BA-WE2 + C-BA
Thickness (mm)	31	31
Average temperature (ºC)	10.0	10.0
Density (kg/m³)	53.5	52.3
λ (W/m·K)	0.0202	0.0200

6.3.4.4. Normality test

Similar to the previous case, Normality Test results indicate consistent insulating properties for both systems, with slight differences in the $\lambda_{21} - \lambda_0$ value. Slight improvement can be seen with the use of a co-blowing agent.

Table 19. Normality test results for BA-WE2 and BA-WE2 + C-BA systems

	BA-WE2	BA-WE2 + C-BA
Thickness (mm)	19	19
Average temperature (ºC)	10.0	10.0
Density (kg/m³)	52.4	50.8
λ_0 (W/m·K)	0.0200	0.0197
λ_{21} (W/m·K)	0.0251	0.0240
$\lambda_{21} - \lambda_0$ (W/m·K)	0.0051	0.0043

6.3.4.5. Compressive strength

Both products were tested under similar conditions to determine their compressive strength at 10% deformation and corresponding modulus. The system BA-WE2 demonstrated slightly higher compressive strength at 10% strain (613 N vs. 535 N) and stress (0.253 MPa vs. 0.211 MPa), indicating better resistance under mechanical load.

The compressive modulus values are also very similar (4.42 MPa for BA-WE2 vs. 4.51 MPa for BA-WE2 + C-BA), indicating comparable foam rigidity, the observed decrease in compressive strength at 10% strain for the BA-WE2 + C-BA system is consistent with its slightly lower density. Therefore, the mechanical performance test confirms that this recipe change is suitable for structural applications despite the minor reduction in load-bearing capacity.

Table 20. Compressive strength results for BA-WE2 and BA-WE2 + C-BA systems

	BA-WE2	**BA-WE2 + C-BA**
Thickness (mm)	50	50
Area (mm²)	2500	2500
F10% (N)	613	536
σ10% (MPa)	0.253	0.212
Compressive modulus (MPa)	4.42	4.51

6.3.4.6. Small burner fire test

In the small burner fire test, the average fire propagation length remained well below the 150 mm threshold for both systems.

Table 21. Small burner fire test results for BA-WE2 and BA-WE2 + C-BA systems

	BA-WE2			**BA-WE2 + C-BA**		
Samples	3			3		
Test type	Superficial			Superficial		
Flame spread (mm)	110	120	125	120	120	115
Surf. of damage (mm)	90	120	130	110	100	100
Flame duration (s)	20	18	19	8	8	11
Classification	E			E		

6.3.4.7. Open-cell content

The open-cell content analysis also shows comparable results between systems, with BA-WE2 + C-BA maintaining a low open-cell content that, although being higher, satisfies closed-cell foam requirements.

Table 22. Open-cell content results for BA-WE2 and BA-WE2 + C-BA systems

	BA-WE2	**BA-WE2 + C-BA**
Samples	3	3
Specimen density (g/cm³)	0.0525	0.0513
Longest side (cm)	3.92	4.02
Shortest side (cm)	2.05	1.95
Middle side (cm)	3.03	2.97
Open-cell content (%)	2.25	3.37

6.3.5. HF-2

In the HF-2 study, physical tests were carried out on three sprayed panels, each corresponding to a different production batch manufactured at the company's facilities. The tested products were: BA-RF1, BA-WE2 and BA-WE2 + C-BA.

This procedure was conducted as part of the CE marking follow-up process, which requires testing of four distinct production batches (every year) to verify continued conformity with the declared performance characteristics. According to the standard, each batch must be sprayed and tested under controlled conditions.

Thermal conductivity, open-cell content, compressive strength and Normality test (last one only for BA-RF1 system).

6.3.5.1. Thermal conductivity

The BA-RF1 formulation shows the highest thermal conductivity value (0.0217 W/mK), which is consistent with its higher density (72.1 kg/m³). This is due to its use in roofing and flooring applications. The other terms are within the expected values.

Table 23. Thermal conductivity results for BA-RF1, BA-WE2 and BA-WE2 + C-BA systems

	BA-RF1	**BA-WE2**	**BA-WE2 + C-BA**
Thickness (mm)	28.8	28.0	28.0
Mean temp. (°C)	10.0	10.0	10.0
Density (kg/m³)	72.1	51.8	49.9
λ (W/mK)	0.0217	0.0206	0.0202

6.3.5.2. Normality Test

As established by CE marking requirements (EN 14315-1), thermal ageing tests like the Normality test must be conducted at least every two years for declared products. This year was necessary to conduct it for the BA-RF1 formulation. Only the initial thermal conductivity has been measured so far, as the ageing procedure is still ongoing. The sample is currently being stored at 70 °C for 21 days in accordance with the requirements of the Normality test defined by the CE marking standard.

Table 24. Normality test results for BA-RF1 system

	BA-RF1
Thickness (mm)	18.5
Mean temp. (°C)	10.0
Density (kg/m³)	74.6
λ_0 (W/mK)	0.0213
λ_{21} (W/mK)	-
$\Delta\lambda_{21} - \lambda_0$ (W/mK)	-

6.3.5.3. Open-cell content results

All systems exhibit low open-cell content, which is desirable in closed-cell spray polyurethane foams. The observed differences in open-cell percentage between BA-RF1, BA-WE2, and BA-WE2 + C-BA can be attributed to their intended applications, with BA-RF1 being designed for roofing and flooring.

Table 25. Open-cell test results for BA-RF1, BA-WE2 and BA-WE2 + C-BA systems

	BA-RF1	**BA-WE2**	**BA-WE2 + C-BA**
Samples	3	3	3
Density (g/cm³)	0.0730	0.0531	0.0479
Longest side (cm)	4.00	4.00	3.99
Shortest side (cm)	2.00	2.01	1.92
Average side (cm)	3.02	2.99	2.97
Open-cell content (%)	3.17	3.89	4.12

6.3.5.4. Compressive strength

BA-RF1 shows significantly superior compressive strength and modulus compared to the other two formulations, reflecting its higher density and structural robustness. However, both BA-WE2 and BA-WE2 + C-BA maintain acceptable performance for less demanding applications.

Table 26. Compressive strength results for BA-RF1, BA-WE2 and BA-WE2 + C-BA systems

	BA-RF1	**BA-WE2**	**BA-WE2 + C-BA**
Thickness (mm)	50	50	50
Area (mm²)	2500	2500	2500
F10% (N)	1028	641	610
σ10% (MPa)	0.41	0.26	0.24
Compressive modulus (MPa)	9.72	4.01	4.73

6.4. Discussion of experimental results

The discussion is structured by experimental studies, taking into account both reactivity-focused and physical property evaluations. The implications of each set of results are interpreted according to application requirements, cost-performance balance, and CE marking criteria.

6.4.1. HM-1: Formulation optimization for roofing and flooring

In HM-1, the formulation BA-RF2 was developed from BA-WE2 with the goal of providing a cost-efficient alternative to BA-RF1. The proportions of PE-A1 and PE-A2-FR were increased to enhance flame retardancy and crosslinking density, while PES-1, water content and HFO contents were reduced to lower the overall cost and optimize foam density and compressive strength. This new system achieved reactivity and free-rise density profiles comparable to BA-RF1, while enabling a raw material cost reduction of approximately 0.50 €/kg.

6.4.2. HM-2: Partial substitution of HFO for cost optimization

The second study focused on evaluating the partial substitution of HFO with a small amount of co-blowing agent (C-BA) in the BA-WE2 system, with the aim of reducing formulation cost. The selected ratio (7.4% HFO + 0.5% C-BA) allowed the product to maintain a flash point above 36 °C. Although this value does not exceed the 60 °C threshold typically required to declare a material as non-flammable, the system passed the sustained combustibility test, thus enabling its declaration as non-flammable. This modification has since been validated and adopted for regular production, contributing to both compliance and economic efficiency.

6.4.3. HM-3: Performance-cost trade-offs in water-based systems

The HM-3 study involved multiple steps to explore low-cost and enhanced performance alternatives to the W-M system.

Initial reactivity and fire resistance evaluations showed that W-M-X, despite reduced raw material costs, could meet the fire requirements only when not combined with the co-blowing agent. Successive trials tested different silicone stabilizers (SIL-1 to SIL-4), identifying SIL-4 as the best-performing option in terms of flame spread and burn duration. Based on these results, the formulation W-M-LW + C-BA with SIL-4 was selected for long-term ageing evaluation at 45 °C, where its reactivity, density, and foam appearance are being monitored over three months to assess thermal stability.

6.4.4. HF-1 and HF-2: Physical characterization of projected foams

The HF studies focused on the physical characterization of projected spray polyurethane foams to ensure regulatory compliance and monitor product consistency over time.

In HF-1, the purpose was to evaluate the properties of foams derived from BA-WE2 and its modified version containing C-BA, following the formulation adjustments defined in HM-2. These tests allowed verification that the modified system continued to meet performance expectations under CE marking requirements.

In HF-2, three different commercial batches (of SPF systems BA-RF1, BA-WE2, and BA-WE2 + C-BA respectively), were assessed to update the Declaration of Performance (DoP) and maintain conformity with the CE marking scheme. A decrease in density was achieved for the formulation BA-WE2, along with a slight improvement on thermal conductivity. In line with EN 14315-1, the normality test was conducted for the BA-RF1 product, because every two years a declared system is subject to periodic thermal conductivity (under ageing) monitoring. The rest of the tests (compressive strength, dimensional stability, thermal conductivity, small burner reaction, and open-cell content) confirmed the reliability and suitability of all systems for their intended applications.

7. Conclusions

This work has addressed the optimization of spray polyurethane foam (SPF) systems by evaluating cost-effective formulations that maintain the required technical performance. The study included both reactivity-oriented tests and physical characterization of projected foam panels, aligning with CE marking regulations and industrial application needs.

First of all, the development of BA-RF2 from BA-WE2 proved successful, achieving comparable reactivity and density to BA-RF1 while reducing raw material costs by approximately 0.50 €/kg. This suggests that the optimization strategy can deliver economic benefits without compromising essential properties such as compressive strength or flame behaviour.

The partial replacement of HFO with C-BA in the BA-WE2 formulation, as explored in HM-2, maintained adequate flammability performance (flash point and sustained combustion) while lowering the formulation cost, as it enabled the partial replacement of a high-cost blowing agent. This led to the product's approval and ongoing production.

HM-3 highlighted the complexity of cost-performance trade-offs in water-based systems. Although initial cost-reduction and enhanced performance attempts (e.g., W-M-X with C-BA) did not meet fire resistance requirements, the use of alternative silicone stabilizers—especially SIL-4—offered improved performance. The system W-M-LW + C-BA with SIL-4, that was intended for reducing costs and improving adhesion to the substrate at low temperatures, entered a thermal ageing program, providing insight into long-term foam stability under elevated temperatures. Hopefully, this will led to a recipe change validation and implementation in regular production.

The HF studies provided a physical evaluation of the developed systems. All tested products (BA-RF1, BA-WE2, and BA-WE2 + C-BA) complied with CE performance criteria, including compressive strength, thermal conductivity, dimensional stability, open-cell content and flame behaviour.

Overall, the results demonstrate the feasibility of reducing the cost of SPF systems without compromising compliance or performance, through the full or partial substitution of high-cost raw materials such as polyols and blowing agents. The optimized systems developed and evaluated in this work can contribute to more sustainable and economically viable building materials, meeting the expectations of modern construction and insulation standards.

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