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# Investigating the Use of Supercritical Fluids in the Production of Polymer Sponges

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## Abstract

Supercritical fluid technologies provide the possibility of obtaining new products with special features and designing new processes that are environmentally friendly and sustainable, and therefore, in this article, the important principles and basics of the production of sponges from a variety of polymer materials along with factors Affecting the properties of the obtained product (such as thermodynamic and rheological properties), it is briefly reviewed and analyzed. Also, in this article, the position of this technology is also mentioned. Therefore, the knowledge of this technology provides the possibility of better understanding of the processes and their proper application by researchers and researchers for the purpose of use in industrial dimensions and scale.

**Keywords:** Foams, Supercritical Fluids, Polymers

## Introduction

Sponges are the simplest and most ancient multicellular organisms. These creatures lack any mechanical defense structures, and thus, over the years, they have evolved the ability to produce secondary metabolites as a form of chemical defense to ensure their survival. One of the most significant applications of these secondary metabolites is the use of their biologically active chemical compounds as pharmaceuticals (Nazemi et al., 2015). Today, supercritical fluid technology is rapidly growing and expanding across various fields of science and engineering. In fact, supercritical fluids, especially carbon dioxide, are used in industries such as composites, polymers, pharmaceuticals, food, and even nuclear technology, in various applications such as energy, extraction, solubility, nanoparticle production, polymerization, polymeric foam production, and more (Sediqian & Tabibzadeh, 2022). The term "foam" refers to a class of multiphase materials in which one phase is in a gaseous state. These materials can be found in nature in various forms, from bones to tree trunks, and are visible in everyday life, from cars to the frames of beds. The widespread use of these materials is due to the properties derived from

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their cellular structure, which grants them a wide range of capabilities. For instance, as thermal insulation, the presence of a network of closed cells in their structure acts like tiny double-pane units, preventing heat transfer from one cell to another. Additionally, the small size of these pores is necessary to reduce thermal conductivity. Conversely, when these materials are used as sound absorbers, the cells must be interconnected, allowing open-cell structures to facilitate the entry of sound waves into the foam and their subsequent propagation through repeated reflection. When these materials are used as scaffolds in tissue engineering, it is essential that the pores be interconnected to allow for the passage of nutrients and waste materials, and that they be large enough to facilitate proper communication and proliferation between living cells.

In the present era, with population growth and industrial development, noise pollution, which is the source of many social and urban disorders, is increasing. Moreover, noise pollution causes fatigue and decreases productivity in work environments. Sound is a type of mechanical wave that requires a medium for transmission. Polymers, due to their viscoelastic properties, particularly in the viscous regions, dissipate and absorb sound waves through viscous damping. A review of the literature shows that adding nanoparticles improves the properties of polyurethane foams, particularly their acoustic characteristics. These nanoparticles increase the probability of sound wave attenuation by enhancing the pathways for sound propagation through the polymer sample. Based on their aspect ratio, nanoparticles are classified into different types, including sheet-like nanoparticles such as graphene and graphene oxide, tubular nanoparticles like carbon nanotubes, and particulate nanoparticles such as metal oxides like zinc oxide and nanoclays. Many researchers have investigated the effect of each of these nanoparticles on sound absorption in polyurethane foam samples (Langroudi & Abbaszadeh, 2021). Among the various compounds used as physical blowing agents, supercritical fluids, especially supercritical carbon dioxide, hold a special position due to their adjustable pressure capabilities and their diverse applications in processes such as extraction, solubility, nanoparticle production, polymerization, saturation, and more, in industries ranging from pharmaceuticals and food to polymers and nuclear technology(Sodeifian et all,2023& Sodeifian et all,2018& Daneshyan & Sodeifian,2022& Fathi & Sodeifian,2022& Sodeifian et all,2018 & Liu et all,2020 & Sodeifian et all,2019 & Sodeifian & Sajadian,2019 & Sodeifian et all,2022). The increasing tendency to use carbon dioxide is due to its lesser environmental impact compared to traditional blowing agents like hydrochlorofluorocarbons (HCFCs) and its higher safety compared to flammable hydrocarbons like pentane. Therefore, this study examines the application of supercritical fluids in the production of polymeric foams.

## Research Background

Azimi (2023) conducted a study titled *Cellular Structures of Polyvinyl Alcohol Sponges Prepared by the Discontinuous Method*. In this study, the sponging process of polyvinyl alcohol with the addition of certain compounds using the discontinuous process was investigated. The presence of a crystalline structure in polyvinyl alcohol is attributed to the strong hydrogen bonds between its chains. By adding certain plasticizers and additives, these strong bonds can be broken, and new bonds can be formed. Studies show that the sponging ability of the semi-crystalline bio-polymer polyvinyl alcohol has significantly improved after adding plasticizers and nanoparticles as nucleating agents. Various analyses revealed that the appropriate intermolecular interactions in polyvinyl alcohol and additive blends, in turn, extend the processing window of polyvinyl alcohol. Due to its non-toxicity and high biocompatibility, polyvinyl alcohol is an excellent candidate for tissue engineering applications. Considering this, examining the morphology of the produced sponges using scanning electron microscopy and studying the cellular structures of the resulting sponges is of particular importance. It was found that significant changes in the structure and cellular density of polymeric sponges could be observed by altering the operational variables and adding different polymeric compounds. Furthermore, the cell size distribution, cellular density, and porosity of the produced sponges can be controlled by using specific amounts of additives and adjusting pressure and temperature (Azimi, 2023).

Nasiri and colleagues (2022) conducted a study titled *Antimicrobial Activity of Bacteria Associated with the Marine Sponge Haliclona sp. Collected from the Persian Gulf*. This study aimed to determine the antimicrobial activity of bacteria associated with the marine sponge Haliclona sp. collected from the Persian Gulf. Methods: In the present study, the antimicrobial effects of bacteria associated with the marine sponge Haliclona sp. collected from Larak and Kish Islands in the Persian Gulf were studied against the gram-positive bacterium Micrococcus luteus. After identifying and isolating the samples, the associated bacteria were purified. Isolation and purification were carried out on Starch casein nitrate agar, Marine Zobell agar, Glycerol asparagine agar, and Marine sponge agar media. Nutrient broth was used for screening the biological activities of the purified bacteria. Finally, the antimicrobial activity of the isolated bacteria was measured using the well diffusion method against the gram-positive bacterium Micrococcus luteus. Results: The highest number of bacteria associated with the marine sponge Haliclona sp. was isolated from the media MSA, SCNA, MZA, and GAA, with CFUg-1 values of 5.88±0.05, 4.87±0.05, 4.20±0.03, and 2.75±0.23, respectively, from sponges collected from Larak Island, and CFUg-1 values of 6.15±0.07, 5.08±0.05, 4.90±0.07, and 4.19±0.19, respectively, from sponges collected from Kish Island. A total of 121 bacterial strains were purified, of which 12 strains exhibited antibacterial activity. Conclusion: The present findings showed that the marine sponge Haliclona sp. has significant antibacterial effects, making it a suitable candidate for future studies on isolating effective antibacterial compounds (Nasiri et al., 2022).

Souri Nejad and colleagues (2022) conducted a study titled *Antifungal Effects of Steroids from the Persian Gulf Sponge Axinella sinoxea Alvarez & Hooper, 2009*. The purpose of this study was to investigate the antifungal effects of steroid compounds extracted from the Persian Gulf

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sponge Axinella sinoxea. For this purpose, extraction was performed on sponges using a range of solvents with different polarities, and fractionation was carried out using a silica gel chromatography column. Steroids in the fractions extracted from the silica gel column were identified using thin-layer chromatography and gas chromatography-mass spectrometry. The antifungal properties of the steroid-containing fractions were studied using the tube dilution method to determine the minimum inhibitory concentration and minimum fungicidal concentration against Aspergillus fumigatus and Candida albicans. The steroids identified from the sponge included Stigmasta, Ergosta, Cholest, and Norgorgosta compounds, which showed varying results in inhibiting the growth and killing the fungal and yeast strains tested at different concentrations. Stigmasta and Ergosta compounds exhibited better antifungal effects compared to other steroids, with Ergosta showing the best inhibitory and fungicidal effects against the yeast. In summary, promising results were obtained regarding the antimicrobial properties of steroid compounds extracted from the A. sinoxea sponge from Larak Island in the Persian Gulf, revealing the necessity for further research into the synthesis of marine bioactive compounds for potential drugs (Souri Nejad et al., 2022).

Mohammadi and colleagues (2022) conducted a study titled *Design and Fabrication of Polyurethane Sponges Reinforced with Rock Wool Fibers: The Effect of Reinforcement Amount on Acoustic and Mechanical Properties*. This study focused on improving the acoustic and mechanical properties of polyurethane sponges modified with various percentages (0-1.2%wt) of rock wool fibers synthesized during the polymerization process. Methods: To construct acoustic composites, the relationship between non-acoustic parameters (airflow resistance, porosity, density, and fiber reinforcement percentage) and microstructure with the sound absorption coefficient was examined. The sound absorption coefficient was measured using a two-microphone impedance tube in the frequency range of 63Hz to 6400Hz in accordance with ISIRI 9803 without an air gap behind the sample. The physical structure morphology and tensile strength were examined by field emission scanning electron microscopy (FE-SEM) and tensile strength testing, respectively. Non-acoustic parameters, including porosity and airflow resistance (AFR), were measured using porosimetry testing and the Archimedes method. Results: The findings showed that increasing the percentage of rock wool fibers in the polyurethane sponge increased the surface hardness of the sponge, and the sound absorption coefficient increased across the entire frequency range for all studied samples. The highest sound absorption coefficient was observed in polyurethane-fiber composite sponges with 1.2%wt rock wool fibers in the frequency range of 2000-4000Hz. This increase in sound absorption coefficient is likely related to the reduced pore size with increased fiber content, as shown by the morphology results. The mechanical properties (tensile strength) of the sponges were also studied by varying the amount of reinforcing fibers. The results indicated that the tensile strength of the composite sponges significantly improved with the addition of fibers. Finally, regression analysis was conducted to examine the relationship between

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non-acoustic parameters (including airflow resistance, porosity, density, and fiber filler content) and the sound absorption coefficient, showing a relatively good fit between experimental and statistical data. The results of this study demonstrated that these composite sponges can be used for sound reduction purposes (Mohammadi et al., 2022).

Mahran and colleagues (2020) conducted a study titled *Numerical Investigation of the Melting Process of a Non-Newtonian Fluid in a Metallic Foam*. The non-Newtonian behavior of a phase change material (PCM) inside a coaxial porous tube was studied using a deforming mesh technique. The inner and outer tubes were subjected to high and low temperatures, Th and Tc, respectively, while the lower and upper surfaces were thermally insulated. The finite element method (FEM), implemented in an optional Eulerian-Lagrangian moving mesh technique (ALE), was used to solve the weak forms of the governing equations. Stefan conditions were applied to track the solid-liquid interface of the PCM during the melting process. Mesh independence tests were conducted, and validation of the results was performed by comparing them with several experimental cases published in the literature. Simulations indicated that increasing the Stefan number significantly improved the melting rate. As the Stefan number increased from 0.014 to 0.01, the non-dimensional time for complete melting decreased from 1.313 to 0.937. Additionally, a sharp increase in the melting rate was observed when decreasing the power-law index. When the power-law index decreased from 1 to 0.6, the complete melting time subsequently decreased by 54% (Mahran et al., 2020).

Derakhshesh and colleagues (2012) conducted a study titled *The Role of Artificial Structures in Increasing Sponge Species Diversity in the Northwest of the Persian Gulf (Bahrakan Coast)*. This study was conducted to investigate the role of artificial structures in the development of sponge communities in artificial habitats constructed on the Bahrakan coast, located in the northeastern Persian Gulf. Sampling was carried out over four seasons, from spring 1388 to late winter 2009. A total of four stations were selected for sampling, with one station at the site of older structures (station D) and three stations at the site of newer structures (stations A, B, and C). Samples were randomly collected by placing 25x25 cm quadrats at a depth of 12 meters. The results showed that artificial habitats, by increasing the heterogeneity of the substrate, led to an increase in species diversity in the environment, and after a decade since their construction, there has been a significant increase in sponge species diversity in the area. Therefore, similar structures to the current ones can provide a suitable habitat comparable to natural hard substrates (Derakhshesh et al., 2012).

## Current Status

The production of foams using supercritical fluids is a field marked by challenges in research and development. In the past, comprehensive review articles have been published covering various

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aspects of this field (Chauvet & Sauceau,2017 & Tsivintzelis et all,2016 & Kiran , 2019). These include topics such as the formation of nanocellular foams (Forest , 2015), foam production from polymer blends (Wan et all, 2017), foam production from copolymers and semi-crystalline polymers (Mi et all,2016), elastomers (Meng et all,2017), composites, and high-performance engineering plastics (Cafiero & Iannace,2016), as well as biodegradable and non-biodegradable foams (Ganjyal et all,2004). In this article, we present the general principles of foam production and briefly describe common processing methods and polymer material properties that become important during foaming.

Among supercritical fluids, carbon dioxide gas is the primary fluid used as a physical blowing agent in the production of polymer foams. Although other gases, such as nitrogen, are sometimes used in combination with carbon dioxide. The foam production process involves two main stages: (a) absorption or dissolution of carbon dioxide under pressure into the polymer matrix to form a solution of polymer melt and gas, and (b) nucleation and growth of bubbles, which occur due to a decrease in pressure or an increase in temperature. The growth of bubbles and the subsequent foam formation continue until mass transfer is halted by polymer crystallization or vitrification. The occurrence of crystallization or vitrification prevents the merging of cells, thereby stabilizing the structure (Cafiero & Iannace,2016). In cases where vitrification is delayed, depending on the cell growth rate and the type of polymer material, cell wall deformation leads to the collision of adjacent cells, tearing of walls, and eventually cell merging. The merging of a large number of cells may even lead to the collapse of the entire foam structure (Park et all,1998). In general, it can be said that the enlargement of pore size due to cell merging or the compression of the overall foam shape caused by its collapse results in undesirable properties for the foam.

The structure and pore size of the product are closely related to the following factors: (a) the concentration of the blowing agent, which is limited by the solubility of the gas in the polymer matrix, (b) the molecular diffusion capacity of carbon dioxide in the polymer matrix, (c) the surface tension between the polymer-gas solution in contact with carbon dioxide, (d) how the polymer material's transition temperatures (Tg and Tm or Tc) are altered and how the corresponding processes (such as softening/glass transition and melting/crystallization) are affected, and as a result, how the rheological properties (such as viscosity and modulus) change during the absorption stage and the pressure reduction stage (Kiran , 2019). A rapid pressure drop at constant temperature leads to smaller pore sizes or a higher cell density in the foam. Similarly, if gas concentration and pressure drop rate remain constant, higher temperatures result in larger pores, and in such conditions, the overall foam density initially decreases and then increases (Kiran,2010).

The properties of the resulting product depend on the following: the temperature and pressure during the absorption stage, the pressure reduction rate during the second stage, the diffusion

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coefficient of carbon dioxide under the system's temperature and pressure conditions, the thermophysical state of the polymer system (e.g., the proximity to the melting or glass transition temperature when the system is in a specific state such as crystalline, rubbery, or glassy), and the rate of rheological property changes in the polymer material (e.g., viscosity and modulus).

In general, the three common operational methods in foam production are: batch operations, foam production using extrusion processes (with the help of a physical or chemical blowing agent), and injection molding operations. All three methods rely on the polymer material's ability to form a pressurized solution of polymer and gas, and the possibility of separating them through pressure reduction or temperature increase, which leads to nucleation and bubble growth.

Batch operations involve placing a polymer sample in a device under high-pressure carbon dioxide for a specified duration (this duration allows for absorption, and its length depends on the bidirectional diffusion of carbon dioxide and polymer) and then reducing the pressure (during which gas bubbles form due to the pressure reduction). If the absorption stage is performed at a relatively low temperature, the polymer matrix may seem too rigid for foaming during the pressure reduction stage. In this case, the polymer matrix, along with the trapped carbon dioxide, can be removed from the device in solid form, and then heated to soften and enable nucleation and foam formation. The foam production process using this method is a two-stage process known as solid-state foaming.

Batch processes are widely used in research settings (usually with small samples, around a few grams, in the form of a circular disc) because they are relatively simple to perform and allow for precise control of process variables such as temperature, pressure, and time. However, the downside of this process is its low efficiency, which limits its application in industries that prioritize high-speed production. The demand for high efficiency is met through the extrusion process. In this method, carbon dioxide is injected into the extruder and mixed with the polymer melt under high pressure. The solution of polymer and carbon dioxide undergoes a rapid pressure drop upon exiting the device, which leads to carbon dioxide diffusion and associated nucleation. For large-scale foam production, the extrusion method is preferred due to its compatibility with continuous production processes and easier scalability. Nonetheless, foam production using this method also presents specific challenges that will be discussed further: (a) the limitation of pressure at the carbon dioxide injection point created by the extruder body, (b) the time available for carbon dioxide absorption or dissolution in the polymer material before it exits the device, and (c) having sufficient melt viscosity in the polymer, which is crucial to prevent carbon dioxide diffusion and the collapse of the foam structure after exiting the device.

It is noteworthy that in the extrusion process, carbon dioxide gas acts as a softening agent, reducing the operating temperature and improving the melt's rheological properties, while also playing a

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role in expanding the polymer material as it exits the extruder (Chauvet & Sauceau,2017 & Tsivintzelis et all,2016 & Kiran , 2019& Forest , 2015& Wan et all, 2017). In practice, this process is usually carried out using single-screw or twin-screw extruders or machines composed of two extruders that are connected sequentially. As the polymer melt passes through the die and exits the device, the inevitable diffusion of carbon dioxide due to the pressure drop limits the degree of expansion (or the degree of foaming). This creates a significant problem when using polymers with low melt strength. To address this issue, a common solution is to freeze the outer (surface) layers by lowering the temperature inside the die. However, this approach can be challenging for producing foams with dense cells, as the polymer's rheological properties must allow for bubble growth while also preventing cell merging. For example, foam production from polyolefins (such as high-density polyethylene, low-density polyethylene, and isotactic polypropylene) is difficult due to their low melt strength, which must resist cell growth (Wan et all, 2017). To apply the aforementioned approach, a practical method that has gained attention is increasing the carbon dioxide absorption rate in the extruder to reduce the melt viscosity during extrusion at low temperatures. In this way, the melt will have sufficient strength when exiting the die. However, injecting large amounts of carbon dioxide into conventional extruders, which have been modified for foam production using carbon dioxide, presents challenges. Thus, a new generation of extruders specifically designed for foam production with carbon dioxide is necessary.

Injection molding has gained attention as an economical and feasible method for producing foams with complex three-dimensional geometries. This technology is rapidly advancing to improve performance (Egger et all,2005), particularly in addressing the problem of premature foaming during injection (before the mold and within the runner and sprue). For instance, to reduce premature foaming and limit the degree of foaming, reverse injection molding technology has been developed. In this process, the mold cavity is initially filled with the polymer material, and after a residence time, part of the mold is suddenly and appropriately displaced. The gas dissolved in the polymer material experiences a significant pressure drop as the cavity expands, initiating uniform foam formation throughout the molded product. Another different method to mention is gas-filled bead injection molding, where polymer pellets filled with gas are first foamed and then formed into beads via extrusion (Sun & Turng,2014). These gas-bearing polymer beads are used as polymer feedstock in injection molding systems to produce three-dimensional foam parts.

### Type of Polymer Used and the Key Role of Its Properties

Foam production using carbon dioxide is generally performed with amorphous and glassy polymers, particularly polystyrene, which has been widely used for foam manufacturing. Foam production from semi-crystalline polymers is more challenging due to the very limited operational framework. The upper limit is restricted by insufficient melt strength at high temperatures, while the lower limit is constrained by inevitable crystallization at lower temperatures (Di Maio &

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Kiran,2018). The critical properties that a polymer material must possess for foam production using gases include: a) acceptable solubility of the blowing agent, b) sufficiently high melt strength, and c) the availability of methods to stabilize the material around the operational framework (through crystallization or glass transition).

One noteworthy phenomenon that can assist the foaming process is the softening of the polymer material due to the blowing agent, which is particularly evident with carbon dioxide (Chauvet & Sauceau & Kiran , 2019). For example, when polystyrene is exposed to carbon dioxide at a pressure of 50 bar under ambient conditions, its glass transition temperature decreases by approximately 50°C. Similarly, the melting temperature of poly(ε-caprolactone) decreases by about 25°C when exposed to carbon dioxide at 100 bar. As the polymer melt and carbon dioxide solution exit the extruder, the release of carbon dioxide and the corresponding reduction in softening effects (equivalent to a cooling stage) significantly contribute to the foam's stability.

For foam production from a semi-crystalline polymer, Figure 1 demonstrates how the operational framework (within the temperature range between Tg and Tm) is broadened when softening occurs in the presence of carbon dioxide. The figure illustrates the importance of process temperature relative to the actual values of Tg and Tc in the presence of carbon dioxide, where the temperature differences (T-Tg) and (T-Tc) determine the rate of glass or crystalline formation in the polymer material. These temperature differences affect the rate of cell growth and the durability of the polymer. In amorphous polymers, where Tc (or Tm) does not appear, the Tg value decreases. Following the reduction in Tc, the required processing temperature for the melt becomes lower, expanding the operational framework from Tc0 to TcP, which is critical for foam production using extrusion (Di Maio & Kiran,2018).

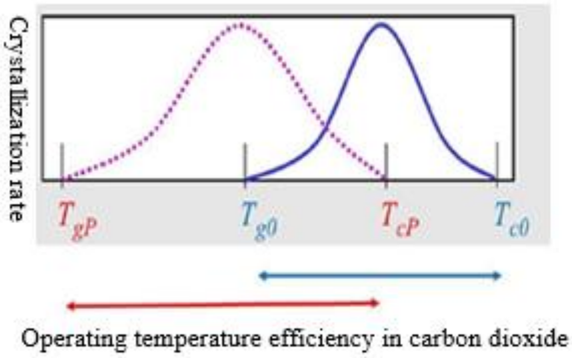

Figure 1:

Reduction of the crystallization temperature (Tc0) or melting temperature and glass transition temperature (Tg0) of a polymeric material at ambient pressure and in the presence of carbon

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dioxide, which dissolves under pressures TcP and TgP. The operational temperature range expands due to the presence of carbon dioxide. The operational framework of the process extends for both solid-state foaming and melt-extrusion foaming of a polymeric material (Di Maio & Kiran,2018).

The viscosity of the polymer matrix also undergoes changes alongside these transformations, which occur following the absorption and release of carbon dioxide (i.e., decreasing during the absorption phase and increasing during the release phase). The viscoelastic properties of the polymer matrix in the presence of dissolved carbon dioxide play a significant role in the final outcomes. During cell nucleation and growth, the melt viscosity of the polymer should be sufficiently low to allow expansion, yet sufficiently high to prevent the leakage of carbon dioxide through the outer cell walls; otherwise, the cells may collapse. The operational temperature and pressure also play a crucial role in the foaming process of semi-crystalline polymers, as they not only affect the regions where the foaming process is completed but also similarly impact crystalline regions and the crystallization rate, which can influence the stability of the produced foam.

Figure 2 shows a schematic representation of the factors that interact during the foam formation process of semi-crystalline polymers. The crystallization temperature (Tc) and, consequently, the stiffness of the polymer matrix change depending on the amount of carbon dioxide absorption, which itself is dependent on temperature and pressure. If the Tc value decreases significantly and the (Tfoaming-Tc) value is large, the matrix stiffening will occur slowly, leading to the widespread diffusion of carbon dioxide out of the foam and thus contributing to its densification. If the temperature is not sufficiently high and the modulus or viscosity of the polymer matrix is high, the molecular diffusion of carbon dioxide into the matrix will occur slowly, leading to limited cell growth and, ultimately, the formation of closed-cell structures. Matrix stiffening happens rapidly before the foam fully expands.

If the temperature during the foaming process is relatively high, the molecular diffusion and widespread penetration of carbon dioxide into the matrix will occur, leading to the formation of larger pores and a higher expansion ratio. However, if the processing temperature is too high, substantial gas leakage into the surrounding environment will cause the cells to collapse, shrink, and condense unless the matrix stiffens rapidly. The challenge is to identify the optimal processing conditions to achieve the desired expansion ratios, as Figure 2 shows that this ratio reaches a maximum along with the temperature of the foaming process (Naguib et all,2004).

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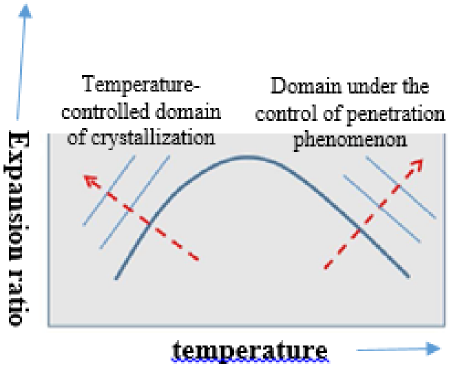

**Figure 2:The effect of crystallization temperature and carbon dioxide permeability as interacting factors influencing the expansion ratio (Naguib et all,2004).**

## Conclusion

A review of the articles and the latest findings in supercritical fluid science and technology demonstrates that this robust scientific field has permeated all sectors of science and engineering. Novel, diverse, and noteworthy information is continuously being published in this area. By specifically reviewing and focusing on the principles of supercritical fluid technology for the production of polymeric foams, our understanding of this field has deepened, enabling the development of this technology for industrial applications. Undoubtedly, research in this scientific domain will open new horizons for scientists and engineers.

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