Sodium Bicarbonate and Citric Acid as Blowing Agents in Polymer Composites with Recycled Textiles: An Alternative to Azodicarbonamide

Marcus Vinicius Badaró de Oliveira Ribeiro[1*], Adriano Puglia Lima[1], Maria Luiza Marques Rapold Souza[2], Pollyana da Silva Melo[1]
[1] *Centro Integrado de Manufatura e Tecnologia – SENAI CIMATEC, Salvador, Bahia, Brasil*
[2] *Universidade SENAI CIMATEC, Salvador, Bahia, Brasil*
marcus.oliveira@fieb.org.br; marcus.vinicius.badaro@gmail.com

Abstract: The study evaluated the influence of sodium bicarbonate (SB) combined with citric acid (CA) as a blowing agent in polymeric compounds produced from recycled polymer and textile waste, comparing its performance with the conventional agent azodicarbonamide (ADC). The compounds were processed in a thermokinetic mixer (DRAISS Homogenizer), and the specimens were prepared by hydraulic press molding, using formulations with 5% and 10% of each blowing agent. Density measurements showed that the SB + CA combination, at a 10% concentration (FCSB10), achieved a 19.29% reduction in density compared to the non-expanded reference, surpassing the applications with azodicarbonamide. Although ADC was slightly more effective at lower concentrations (5%), the combined sodium bicarbonate and citric acid system demonstrated expansion without the need for initiators and without exposure to toxicological risks. The results indicate that this system may be a viable and sustainable alternative to ADC for producing expanded polymers, although adjustments in processing parameters and optimization are still required.
Keywords: Polymer waste. Textile waste. Blow agent. Citric acid. Sodium Bicarbonate.

1. Introduction

Expanded polymers play a significant role in various industrial applications, from packaging and automotive components to thermal and acoustic insulators [1]. Properties such as low density, high strength-to-weight ratio, and excellent energy absorption capacity are achieved by creating a cellular structure within the polymer matrix [1,2]. This process is often carried out with the aid of chemical blowing agents, which decompose under heating to release gases and form the desired cellular morphology [2].

Processing with chemical blowing agents employs the same equipment used in conventional extrusion and utilizes up to 10% by weight of the foaming agent. This process requires precise temperature control and preferably uses a screw with a mixing section [3]. The blowing agent decomposes in the plasticization zone, and when the product is released from the die, expansion occurs to form a cellular material. Typically, the density of foams produced by this method is above to 0.25 g/cm³. The decomposition residues of chemical blowing agents can adversely affect material properties, as they act as fillers [3].

Among the available blowing agents, azodicarbonamide (ADC) has been widely used for decades due to its high gas-yielding efficiency and ability to generate homogeneous, closed-cell structures in a wide range of polymers, such as PVC, polyethylene, and rubbers [2,4]. Its high decomposition temperature approximately 200°C can be adjusted with activators to match the polymer's processing temperature, which has established ADC as a standard additive in the plastics processing industry [5].

However, concerns regarding its use are associated with the byproducts generated during thermally induced decomposition [5,6]. Studies shown that ADC may produce potentially harmful compounds, such as semicarbazide (SEM), particularly in food-contact applications due to its toxicity, as well as the release of urea and carbon monoxide, raising additional occupational and environmental safety concerns [5–8]. Workers exposed to azodicarbonamide exhibited a higher prevalence of respiratory symptoms (coughing, wheezing, chronic bronchitis) and airway irritation, suggesting direct adverse effects from the compound or its decomposition byproducts [9–11].

Given this scenario and the growing demand for safer and more sustainable materials and processes, researchers have sought viable alternatives to azodicarbonamide that perform the same function without generating toxic residues [2]. Promising alternatives have emerged in systems based on sodium bicarbonate (SB), due to its ability to release carbon dioxide (CO_2) when heated, and citric acid, which decomposes to release gases that assist in cellular structure formation [12].

Several researchers have demonstrated the feasibility of using citric acid (CA) and sodium bicarbonate (SB) as blowing agents in various polymeric matrices, highlighting their significant advantages over azodicarbonamide, particularly in terms of safety and environmental profile [13–15].

Being an exothermic agent, azodicarbonamide operates at higher temperatures (170–200 °C), whereas the SB/CA mixture is endothermic and composed of substances classified as non-toxic and non-hazardous. This eliminates concerns related to handling hazardous chemicals and mitigates environmental impacts [16]. Furthermore, SB and CA masterbatches release a substantial amount of gas (water vapor and CO_2) upon reaction, within a broad temperature range (160–210 °C) compatible with many polymers, enabling the production of high-quality foams at low loadings (1–2 wt%) [16].

This combination of safety, efficient performance, and reduced environmental risk positions the sodium bicarbonate/citric acid system as a superior and more sustainable alternative to traditional blowing agents such as azodicarbonamide.

Extensive studies highlight the critical need to optimize processing conditions, temperature and heating rate, to maximize expansion efficiency and final material properties. Achieving consistent performance across different polymer matrices remains a technical challenge.

Within this framework, the present study aims to investigate the effect of sodium bicarbonate combined with citric acid in recycled polymer waste applications, with direct comparison to azodicarbonamide for producing expanded polymer sheets. This research seeks to contribute to developing safer and more sustainable solutions for the expanded polymer industry.

2. Experimental

Materials

The study was used a waste-derived polymer compound, from polymer waste and textile waste, as the matrix material, incorporating two different chemical blowing agent systems for comparison: (1) azodicarbonamide and (2) citric acid combined with sodium bicarbonate.

Methodology

The samples of the polymer compound were prepared from the formulations contained in Table 1.

Table 1. Composition of Expanded Recycled Composite.

Composition	PWTW	FAZ5	FAZ10	FCSB5	FCSB10
Polymer Waste + Textile Waste	100%	95%	90%	95%	90%
Azodicarbonamide	-	5%	10%	-	-
Citric Acid	-	-	-	2%	4%
Sodium Bicarbonate	-	-	-	3%	6%

In a thermokinetic mixer (Figure 1, MH-Equipment model MH-1000), following initial processing parameters of 800 RPM at low rotation speed with temperature starting from 100°C for feeding the materials (polymer waste, textile waste, and blowing agents). The rotation speed was then increased to approximately 1800-2500 RPM for mixing and homogenization of the formulation, while controlling the mixing temperature until reaching values between 150-160°C to preserve the compound integrity. Upon reaching the designated temperature while maintaining high rotation speed, the composite was maintained for a predetermined time interval to ensure compound homogeneity.

Figure 1. Thermokinetc Mixer (DRAISS Homogenizer)

The molten mass was transferred to a rectangular plate mold mounted on a hydraulic press (Advanced brand, Figure 2) with a 4-ton capacity, preheated to 160°C. The mass was pressed to shape the plate, followed by pressure release to allow expansion while maintaining contact with the heating plates for 30 minutes to enable thermal decomposition of the blowing agents. After 30 minutes, the press was turned off and cooled by natural convection, as the hydraulic press lacked a forced cooling mechanism.

Figure 2. Hydraulic Press

Experimental Characterization

Density Properties

Density testing was performed using a analytical balance, DSL910 (F), which applies Archimedes' principle of buoyancy and displacement to determine density in grams per cubic centimeter.

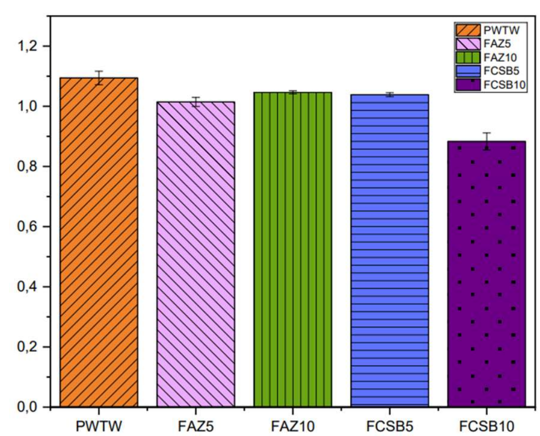
Figure 3. Analytic Balance DSL910

To perform this test, the preparation of specimens with similar dimensions was carried out. Then, a determined volume of anhydrous alcohol with density 0.8165 g/cm³ is added to the container and placed on the balance. The test is performed in two stages. In the first stage, the analytical balance precisely measures the mass (m) of the specimen in air. In the second stage, the specimen was immersed in the anhydrous alcohol, displacing a volume of alcohol equal to its own volume, and by Archimedes' principle, the balance measures the apparent weight of the immersed specimen. The difference between the dry weight and immersed weight (which corresponds to the buoyancy force) is used to calculate the volume of the specimen through Archimedes' principle. Finally, the density of the specimen is calculated by dividing its mass by the determined volume.

3. Results and Discussion

The density evolution of the materials as a function of different blowing agent additions and their respective quantities is presented in Figure 4.

Figure 4. Density behavior graphics.



These results are also presented in Table 2, which allows comparison of the distinct behaviors regarding compound expansion and density reduction.

Table 2. Density Result

Form.	PWTW	FAZ5	FAZ10	FCSB5	FCSB10
Mean	1,0941	1,0143	1,0464	1,0386	0,8830
S.D	±0,0223	±0,0151	±0,0050	±0,0066	±0,0283

The comparative performance of formulations containing azodicarbonamide (FAZ) and citric acid combined with sodium bicarbonate (FCSB), at concentrations of 5% and 10% for density reduction, relative to the non-expanded reference composite (PWTW), revealed distinct responses to the expansion process.

Comparative analysis indicated that the FCSB10 formulation achieved the highest density reduction among the conditions studied, with a decrease of 19.29% relative to the reference. This result contrasts with the performance of FAZ5 and FAZ10, which exhibited comparatively modest reductions of 7.30% and 4.36%, respectively. For the FCSB-based composites, increasing the concentration from 5% to 10% markedly enhanced expansion performance, with density reductions of 5.07% and 19.29%, respectively. In contrast, the FAZ-based composites displayed an inverse trend, with reductions decreasing from 7.30% at 5% concentration to 4.36% at 10%, suggesting the presence of an optimal concentration threshold for this blowing agent

The direct comparison between materials at the same concentration revealed that FAZ5 was 2.40% more effective than FCSB5, whereas at 10% concentration, the relationship was sharply reversed, with FCSB10 outperforming FAZ10 by 15.62%. This phenomenon indicates that additional factors in their mechanisms of action influence the expanded matrix in different ways, with FCSB exhibiting a performance pattern dependent on higher concentrations within the studied parameters.

It is well established that the effectiveness of azodicarbonamide is directly linked to the use of temperatures above 200 °C or to the presence of initiators that lower the component's thermal decomposition temperature [5]. However, the optimal temperature for this condition is not applicable to the material investigated in this study. Furthermore, no initiators were employed in this work, as the objective was to identify a potential substitute for azodicarbonamide.

The results obtained converge with the data reported in the literature. Sadik et al. [13] developed low-density polyethylene (LDPE) masterbatches containing AC, BS, or their combination, evaluating their thermal decomposition kinetics. Their findings showed that BS follows first-order kinetics, releasing CO_2 and H_2O between 100–180 °C, whereas AC decomposes in two stages (160–270 °C), forming intermediates such as aconitic acid and maleic anhydride. The combination of both agents resulted in distinct decomposition stages, enabling better control of expansion at the high heating rates typical of injection molding processes [13].

Vorawongsagul et al. [14] employed SB as a blowing agent in PLA/PBS composites reinforced with cellulose fiber, produced by extrusion and injection molding. The decomposition of SB generated closed-cell structures with reduced density and enhanced mechanical and thermal properties, demonstrating particular suitability for food packaging applications [14].

In addition, Bettelli et al. [15] investigated citric acid as a multifunctional additive in wheat gluten applications, combining it with ammonium bicarbonate. In this study, AC simultaneously acted as a crosslinking agent and plasticizer, producing low-density foams with high porosity and predominantly closed-cell structures [15]. This contrasts with the findings of Sadik et al. [13], who demonstrated that AC and SB mixtures in masterbatches enable expansion kinetics coupled with well-defined decomposition stages for each component. Taken together, these studies highlight the versatility of CA and SB in applications requiring precise control of expansion, reinforcing their potential as sustainable alternatives to conventional blowing agents [13].

Gouissem et al [16] highlighted that the density results confirmed the efficiency of combining citric acid (CA) and sodium bicarbonate (SB) when compared to the traditional blowing agent azodicarbonamide. While CA alone reduced the density by up to 43% and SB by approximately 48.6%, their mixtures at optimized proportions, particularly the 60/40 ratio, promoted a synergistic effect, achieving reductions close to 50% and leading to finer, more uniform, and regular cell structures. These findings demonstrate that the CA/SB combination not only results in lower densities than those obtained individually but also offers structural advantages over azodicarbonamide-based systems, especially in terms of sustainability and environmental profile [16].

Therefore, the data suggests that the selection of the most suitable material is highly dependent on the concentration used and warrants further investigation into the interactions between the materials and the production parameters applied for the development of optimized formulations. Even with the positive response observed for FCSB, the underlying physicochemical mechanisms behind the observed differences, the behavior of additional parameters, and processing conditions, such as molding, temperature, cost-effectiveness, as well as collateral properties of the formulations should be further explored.

4. Conclusion

The results indicated that the combination of sodium bicarbonate with citric acid shows potential to replace azodicarbonamide as a blowing agent. In this study, 10% concentrations were shown to achieve lower densities than the composites containing azodicarbonamide. Although ADC proved to be slightly more efficient than CA combined with SB at 5% concentrations, the significant advantage of the latter at higher concentrations, without the need for initiators and without the associated risks of azodicarbonamide, suggests that this material could serve as a viable substitution alternative. Nevertheless, the use of this expansion system still requires adjustments and optimizations in both formulations and preparation methods, to better understand the potential for greater expansion and to reduce concentration levels, thereby optimizing costs.

For future research, it is essential to investigate specimen molding methodologies, the influence of mold temperature, forming pressure, and strategies to optimize the quantity of chemical agents, with the aim of improving the expansion performance of the developed composites.

Acknowledge

The authors gratefully acknowledge the Materials Characterization Laboratory at SENAI CIMATEC for providing access to the equipment, and ECOLOY for supplying the waste materials used in this study

Reference

[1] Krishnan, V. G., Fiorucci, L., Sarbu, A., & Drenckhan-Andreatta, W. (2025). Characterizing the foaming process of polymers: Review of experimental methods. *Advances in Colloid and Interface Science*, 344, 103579.

[2] Jin, F. L., Zhao, M., Park, M., & Park, S. J. (2019). Recent trends of foaming in polymer processing: A review. *Polymers*, 11(6), 953.

[3] Rabello, M. (2000). *Aditivação de polímeros*. Artliber.

[4] Lendvai, L., Degosy, G., Jakab, S. K., & Fekete, I. (2024). Foam injection molding of poly(lactic acid) with azodicarbonamide-based chemical blowing agent. *Engineering Proceedings*, 79(1), 44.

[5] Guo, R. L., Liu, S. H., & Lu, Y. M. (2023). Investigation of how pressure influences the thermal decomposition behavior of azodicarbonamide. *Journal of Loss Prevention in the Process Industries*, 83, 105062.

[6] KRUTKO, Iryna; DANYLO, Iryna; KAULIN, Viacheslav. Kinetics of azodicarbonamide decomposition in the presence of an initiator for obtaining solid foams. *Вопросы химии и химической технологии*, n. 1, p. 26-34, 2019.

[7] Wu, S., Feng, Y., Chen, S., Zhao, Y., Li, C., Deng, J., & Wang, D. (2025). A novel strategy for the detection of semicarbazide in crustaceans by modified QuEChERS coupled with hydrophilic interaction liquid chromatography–tandem mass spectrometry. *Foods*, 14(3), 541.

[8] Xing, L., Sun, W., Sun, X., Peng, J., Li, Z., Zhu, P., & Zheng, X. (2021). Semicarbazide accumulation, distribution and chemical forms in scallop (Chlamys farreri) after seawater exposure. *Animals*, 11(6), 1500.

[9] WHITEHEAD, Lawrence W. et al. Respiratory symptoms associated with the use of azodicarbonamide foaming agent in a plastics injection molding facility. *American journal of industrial medicine*, v. 11, n. 1, p. 83-92, 1987.

[10] Suojalehto, H., Malo, J. L., & Cullinan, P. (2018). The classification of azodicarbonamide (ADCA) as a respiratory sensitiser; adding to the weight of evidence. *Regulatory Toxicology and Pharmacology: RTP*, *94*, 330-331.

[11] Kupczewska-Dobecka, M., Konieczko, K., & Jurewicz, J. (2024). Effect of occupational exposure to ADCA on the incidence of allergic respiratory reactions–a literature review. *Medycyna Pracy*, *75*(5).

[12] MURPHY, John (Ed.). *Additives for plastics handbook.* Elsevier, 2001.

[13] Sadik, T., Pillon, C., Carrot, C., & Reglero Ruiz, J. A. (2018). DSC studies on the decomposition of chemical blowing agents based on citric acid and sodium bicarbonate. *Thermochimica Acta, 659*, 74–81

[14] Vorawongsagul, S., Pratumpong, P., & Pechyen, C. (2021). Preparation and foaming behavior of poly (lactic acid)/poly (butylene succinate)/cellulose fiber composite for hot cups packaging application. *Food Packaging and Shelf Life, 27*, 100608

[15] Bettelli, M. A., Hu, Q., Capezza, A. J., Johansson, E., Olsson, R. T., & Hedenqvist, M. S. (2024). Affects of multi-functional additives during foam extrusion of wheat gluten materials. *Communications Chemistry, 7*(75)

[16] GOUISSEM, Linda; BENMEKIDECHE, Abdelhakim. Contribution to the expansion of low density polyethylene by the use of chemical blowing agents based on sodium bicarbonate and/or citric acid. Polymer Science, Series A, v. 63, n. 5, p. 542-555, 2021.