



Cell morphology, Compressive strength, and Thermal Properties of Epoxy Foams Using Sodium Bicarbonate as Chemical Foaming Agent

Mohamad Saed Hussein^{1,a)}, Teh Pei Leng ^{2,b)}, Abdul Razak Rahmat ^{3,c)}, Firuz Zainuddin^{4,d)}, Yeoh Cheow Keat^{5,e)}

^{1,2,4,5)} Faculty of Chemical Engineering Technology, Universiti Malaysia Perlis, 02600 Arau, Perlis, Malaysia, ¹⁾ Ras Lanuf Gas and Oil Processing Company (Rasco), Ras Lanuf, Libya
 ³⁾Department of Polymer Engineering, Universiti Teknologi Malaysia, Skudai, Johor, Malaysia
 ^{a)} Corresponding author: ben.hussein2009@gmail.com , ^{b)} plteh@unimap.edu.my, ^{c)}
 <u>firuz@unimap.edu.my</u>, ^{d)} razak@cheme.utm.my ^{e)} ckyeoh@unimap.edu.my

الملخص

تبحث هذه الدراسة في كيفية تأثر خصائص راتنج الإيبوكسي ببيكربونات الصوديوم كعامل نفخ ، حيث تم استخدام بيكربونات الصوديوم لتحسين خصائص راتنجات الايبوكسي المتصلدة بالحرارة. تم تحضير عينات رغوة الايبوكسي باستخدام تقنية الخلط. تم اختيار بيكربونات الصوديوم كعامل نفخ نظرًا لأنها رخيصة الثمن وآمنة بيئيًا وتم اجراء الخواص الانضغاطية والحرارية والتشكلية لكل العينات. تم اضافة كميات من بيكربونات الصوديوم بمقدار 5 و 10 و 15 و 20 و 25 phr على التوالي. أظهرت النتائج المعملية قبول وملائمة عينة الايبوكسي عند 15 مع من يكربونات الصوديوم كمامل نفخ نظرًا لأنها رخيصة الثمن وآمنة بيئيًا وتم اجراء الخواص الانضغاطية والحرارية والتشكلية لكل العينات. تم اضافة كميات من بيكربونات الصوديوم بمقدار 5 و 10 و 15 و 20 و 25 phr على التوالي. أظهرت النتائج المعملية قبول وملائمة عينة الايبوكسي عند 15 phr من بيكربونات الصوديم كما أن لها نسبة مسامية ممتازة ، مع وجود صفات ميكانيكية وكثافة متوسطة. يوضح التشكل أن نسبة كبيرة من البنية المسامية تكونت عند مسامية معازة بيكربونات الصوديوم بيكربونات المعامية تكونت عند

الكلمات الدالة: الايبوكسى، الرغوة، حجم المسام، قوة الانضغاط، التحلل الحراري، بيكربونات الصوديوم.

Abstract:

This study examines how the sodium bicarbonate content (SB) effect the epoxy resin properties. As a foaming agent, SB was employed to enhance the characteristics of thermosetting epoxy resin. Epoxy foam samples were prepared using a mixing technique. Because it is cheap and environmentally safe, SB was chosen as the foaming agent. The compression, thermal, and morphological properties were analysed. The amount of SB was adjusted at 5, 10, 15, 20, and 25 phr, respectively. The lab results showed that the SB was selected at 15 phr and had an acceptable content. It also had an excellent porosity, while having medium mechanical and density property. The morphology demonstrates that a large percentage of porous structure was formed at a SB level of 25 phr.

Keywords: epoxy, foam, pore size, compressive strength, thermal degradation, sodium bicarbonate.





1. INTRODUCTION

Epoxy foam's simple and inexpensive foaming process is one of its benefits from an industrial perspective [1]. Epoxy foams are frequently utilized to lighteren and enhance the insulating properties of structural materials. Due to their naturally buoyant behavior, low moisture absorption, and excellent compressive strength, one of their first applications was in marine structures. SB is an inorganic chemical foaming agent that was utilized as a CO2 generator during the curing process to create new materials with low heat conductivity and better foam properties [2]. So far, there are limited studies involved in the usage of this chemical as the blowing agent in epoxy system. It is of the most prevalent and environmentally friendly agents. In able to manufacture additional forming processes, the two-stage heat transfer is important according to Najib (2009), who employed it with a natural rubber system [3]. To reduce thermal conductivity, SB was added to the existing nitrogen gas in elastomeric foam to form CO2. Epoxy foams were evaluated by acid with 5 phr besides various amounts of SB, 0 to 20 phr. Temperature, shear rate, and CO2 quantity, according to Wan Hamad et al. (2012), influence the viscosity of polymer solutions [4]. Nasruddin et al. (2019) claim that adding SB as a foaming agent to NR can release CO2 through the formation of cell structures. The addition of SB to NR increased mechanical properties [5]. According to Fauzi et al. (2015), epoxy foams with a higher proportion of SB have more porosity and form larger cells. Depending on the level of chemical degradation and cure, the processing and final physical properties alter (crosslinking). The amount of foaming agent used can change the density. The foam should be post-cured for many hours to acquire full strength [6]. Leemsuthep et al., (2017) investigated the conductivity properties of CB in microporous epoxy. According to M.S. Hussein et al. (2018), epoxy foam with 15 phr of SB has suitable porosity and form good cells [7].

2. MATERIALS

Clear epoxy (DER 331) a liquid reaction product and Crystal clear epoxy hardener (A062) were used in this study which provided by Euro Chemo-Pharma Sdn Bhd. The foaming agent in epoxy resin was SB. It was provided by Malaysia's Kasihku Marketing Sdn Bhd in Penang. Acetic acid was supplied by Sigma Aldrich Sdn Bhd.

3. PREPARATION OF SAMPLES

Different amounts of epoxy and SB were utilized at 5, 10, 15, 20 and 25 phr, respectively. The ratio of hardener to epoxy was set at 100:60. After mixing SB and epoxy resins thoroughly for 10 minutes at 200 rpm with a magnetic stirrer, the hardener was added, and the mixture was stirred for an additional 10 minutes, acetic acid was added, and the mixture was thoroughly stirred. then the mixture was poured into the $130 \times 110 \times 4$ mm mold and cured for an hour at 100° C in an oven. The cured was cut into different dimension according to the testing.

4. CHARACTERIZATION

In accordance with ASTM D1895, the density of the polymer samples was determined using a gas pycnometer (Model: Micromeritics AccuPyc 1330) in helium medium which used to find porosity. To determine average pore size, optical microscopy was performed using a standard





bench-top Olympus Optical Microscope. According to ASTM D3575, the cube size was 20 \times 20 \times 15 mm, to compute compressive strength. The specimens were tested with a JEOL JSM-6460 LA model and a 10 kV accelerating voltage. SEM specimen's fracture and flexural surface coated with a tiny coating of palladiums. The method of equilibrium swelling is used to determine cross-link density. At ambient temperature, the specimens are swollen in toluene. taken from the solvent, and the toluene on the surfaces is immediately cleaned with tissue paper. On an analytical balance, the specimens were promptly weighed. Cross-link density, Equation can be used to compute the effective number of moles of cross-linked units per unit weight and Flory-Huggins interaction parameter. The technique outlined in ASTM E 1131 was followed for performing the TGA [31].

5. RESULTS AND DISCUSSION

5.1 POROSITY

Figure 1 depicts the influence of SB content on the porosity of epoxy foam. The maximum amount of fluid that can be stored in open cell foams like those employed in this study is determined by the porosity of the foam, which is a measure of the pore spaces in the foam. The porosity of epoxy foam increased when the amount of SB was increased. This result can be explained by an increase in the volume of decomposed gas, and hence the nucleation sites, as SB levels increase to 15 phr. Porous epoxy have an appropriate porosity in the mechanical term at 15 phr SB content, as compared to all content, as stated subsequently. However, for SB content, the foam porosity increased to about 51% at 15 phr, and 6% at 20 phr. In this case, the mass gain owing to the increased number of pores outweighs the mass loss due to the thinner struts. [8]. The greater the porosity, the more porous the matrix phase. The decomposition of SB took place simultaneously with the epoxy matrix crosslinking. As a result, the percentage of porosities increased and the density reduced as the amount of SB increased. According to [9], the density of polymer foam reduces as the porosities increased. According to [10], the porosities rise as the foaming agent is increased because the gas created by the SB induces the pores to merge and form a larger cells, resulting in larger pores and higher valid porosity.



Figure 1. Influence of SB content on the porosity of epoxy foam

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5.2 CELL MOROPHOLOGY ANALYSIS

SEM analysis was used to evaluate the morphological of the epoxy foam's flexural cracked surfaces. Figure 2 illustrates the flexural surface micrographs of the pure epoxy foam at 0 phr,5 phr and 15 phr of SB. Figure 2 (a) displays that the neat epoxy is generally homogeneous, has brittle smooth glassy flexural surface without any phase separation with many tear lines with cracks, indicating the brittle behavior of epoxy and no any significant plastic deformation can be investigated which performed at room temperature. Furthermore, the cracks propagated freely and randomly, indicating a weak resistance to crack initiation and propagation, which could explain the pure epoxy resin's poor toughness. This indicates that the pristine epoxy has a linear elastic characteristic and that the crack propagates in a planar manner under impact stress. Figure 2 (c) demonstrates that at 15 phr, the optimum SB content formed more pores, resulting in the lowest flexural value. This means that as the amount of SB was increased, the flexural properties were lost. Figure 2 (b) shows how the pores and voids in the sample worked as fracture initiation points, reducing polymer deformation more than Figure 2 (a). When the SB content was increased from 5 to 15 phr, the tear lines were also minimized. In the SEM micrograph, there are a few voids to be seen. Existing voids in the epoxy matrix are an unavoidable issue. These voids could operate as stress concentrators, causing the cured epoxy to become brittle.



Figure 2. SEM micrographs of the flexural surface of epoxy foam at (a) 0, (b) 5 and (c) 15 phr of SB





5.3 AVERAGE PORE SIZE

Cell generation, cell development, and cell stabilization are the three stages of the polymer foaming process. Acetic acid reacts with the SB and the epoxy curing at the on time during the production of foam. The results were illustrated in Figure 3. Extra porosity and interconnection between neighboring cells resulted from the increased SB content. As evidenced in the foam morphology of epoxy foam employing the same quantity of epoxy but a different amount of SB. These foams are open cell foams with a large cell size [11]. The pore size increases from 0.083 to 0.19 mm, with an increase in SB content from 5 phr to 15 phr, as shown in Figure 3. Yao et al. (2014), found that at low contents (1 to 3% wt%), temperature and chemical foaming agent content had little effect on the size of the pores [12]. Comparatively, 5 phr has the smallest cell size; while 15 phr to 25 phr possessed larger cell size. This could be attributed to the fact that during foaming in 20 phr, there was an excess of foaming agent, which resulted in increased gas pressure during the early curing stage, when there was less initial crosslink in the epoxy-hardener. As a result, the pores' boundaries will coalesce with one another, resulting in a large cell [11], [13]. Furthermore, the foaming agent was observed to lower the tensile strength of foams due to a decrease in cell wall as a result of increased void volumes (Gu et al., 2014b). It was elucidated that the presence of SB gave rise to smaller pore size. Theoretically, the existence of CO_2 in the foam structure and the improved pore size should reduce the thermal conductivity [14].



Figur 3. Effect of SB contents on the average pore size of epoxy foam

The interconnection between cells is lower in epoxy foam with 5 phr compared with 15 phr. This could be explained by the presence of too much SB during foaming in 15 phr epoxy foam, which resulted in higher gas pressure during the early curing period, when the initial crosslink in epoxy-polyamide was weaker. As a result, the pores' boundaries will fuse with one another,





resulting in a large cell. [11]. The gas disintegrated too much at more than 10 phr of SB, and the green crosslink matrix could not hold the excess expanding gas. More cells coalesce and the size of the cells is less uniform, as seen in epoxy shape memory foam described by [11]. The efficient creation of epoxy foams is dependent on a precise balance of two simultaneous reactions: matrix curing and chemical foaming agent decomposition. If the polymer matrix is cured first, bubbling will not be possible because the polymer matrix will lose its capacity to flow and distort. Bubbles coalesce and even collapse if bubbling happens first without subsequent cure [15].

5.4 COMPRESSIVE STRENGTH

Figure 4 represents the compressive properties of epoxy foams at various levels of foaming agent content. It follows the opposite trend as the porosity, as seen in Figure 1; epoxy foams with lower porosity exhibited higher compressive strengths. Mechanical properties and density decrease as the amount of foaming agent increases. The experimental values can be fitted using a power-law equation in terms of foam density [16]. It is generally known that as porosity increases, compressive strength decrease [8]. As shown in Figure 4, the compressive strengths of foams with SB content of 5, 10, 15, 20, and 25 phr are 6.89, 5.35, 4.13, 3.86, and 3.65 MPa, respectively, when compared to previously reported epoxy foams of similar density values, these values are quite high. As evidenced by this improvement, SB was not just a foaming agent, but also a modification of the as-prepared epoxy foam.



Figure 4. Effect of SB contents on the compressive properties of epoxy foam

Polymer foams have compression characteristics ranging from 1 kPa to 100 MPa, and 0.1 MPa to 10 GPa, respectively in terms of compression strength and stiffness [19]. The compressive strength of epoxy foams was observed to decrease as the SB amount increased. Epoxy foams had a strength of 6.89 MPa at 5 phr, but porous epoxy had a strength of 4.0 MPa at 15 phr under the same curing condition. It can be addressed by a high volume portion of expandable epoxy mixed with a high SB content, resulting in poor compressive strength of the epoxy foams. [21]. To compare epoxy foams with different porosities and analyse the influence of SB





content on the compressive properties of epoxy foam, it was standardized with respect to their foam density. The compressive strength of the epoxy resin without foaming agent was 15.23 MPa. The foam compressive strengths fell from 6.89 MPa to 3.65 MPa as the SB content was increased from 5 to 25 phr. The results revealed a substantial relationship between compressive characteristics and foam density [22], [23].

5.5 CROSS-LINK DENSITY

Figure 5 depicts the influence of SB content on the foam cross-link density. The percentage of swelling is known to be related to crosslink density, the foam has a higher density of cross-link, indicating that it has a lower percentage of swelling in a given solvent [24]. When compared to epoxy foam with 5, 10, 15, 20, and 25 phr of SB, it is obvious that neat epoxy without SB had the maximum cross-link density. The values of cross-link density of epoxy foams tend to decrease as the content of SB to the epoxy increases. The formulations that added 15 to 25 phr to the epoxy resin had the lowest cross-link density values, which were 17×10^{-3} and 15×10^{-3} mol/ cm³, respectively. Epoxy foam with up to 15 phr exhibited the largest swelling % due to the maximum porosities in their structure and the lowest cross-link density, as illustrated in Figure 5. According to Soh and Yap et al. (2004), a lesser degree of crosslinking causes more swelling and hence a larger softening effect [25]. When the polymer is immersed in the solvent, the crosslinking network prevents solvent molecules from establishing sufficient interactions with the entire polymer, preventing the molecules from being carried off into solution.



Figure 5. Effect of SB content on the crosslink density of epoxy foam

When comparing epoxy foam processed with high content of SB content, it is obvious that higher porosity content resulted in decreased cross-link density. According to Zhang et al. [26],





the crosslinking network influences mechanical properties, which explains why lower compressive strength was reported in samples with larger porosities, as previously discussed. Because of the decreased cross-link, the cell size of 20 phr SB foam is 60.52 % bigger than that of 5 phr SB epoxy foam. The reduced cell size of the 5 phr SB epoxy foam results in a lower length to thickness slenderness ratio, which could make the material's cells less prone to buckling and hence increase fracture toughness. Another factor is the fracture path; 5 phr epoxy foam encounters more cells and solid edges, making it a tougher material by nature [27].

4.6 THERMAL STABILITY

Figures 6 and 7 illustrate the TGA curves and derivative thermogravimetric (DTG) of pure epoxy and epoxy foam, respectively. The maximum degradation temperature (T_{max}), which is the highest thermal degradation rate temperature obtained from the peak of DTG thermograms, and the initial decomposition temperature (IDT), which is the initial 5% weight losses temperature (T_5), were chosen as the characteristic thermal parameters.

Figure 6 depicts the thermal stability of epoxy foam with SB contents of 0, 10, and 15 phr. At 310°C, the epoxy foam one step weight loss, according to this data. Thermal degradation of epoxy foam caused the weight reduction. The temperature of weight loss was practically identical between the epoxy foams with 10 and 15 phr of SB, but the epoxy foam with 15 phr, which contained more pores, had higher thermal stability than the epoxy foam with 10 phr of SB. According to the Guohe et. al. (2011), The pore epoxy contact increases the foam's thermal stability and lowers macromolecule activity, delaying the degradation process. According to a similar observation, the improvement could be due to higher porosity of epoxy foam with higher foaming agent content [28]. Williams and his colleagues were observed, with the lowest density showing the most degradation and degradation temperatures decreasing as density increased in the same chemical in the performance polyimide foams [29]. Different stages of the thermal degradation process, while the second step results in burned foam fragments in the range of 300-500°C [1].

At low temperatures, the thermal decomposition of SB occurs in one step. The most of the foaming agent decomposes between 115 and 190°C [30], [31]. Figure 6 shows that when heated to 800°C, cured systems undergo two-stage thermal decomposition and exhibit thermal behavior identical to that of pure epoxy. This indicated that adding SB to the epoxy matrix had no substantial effect on the epoxy matrix's degradation mechanism. Figure 6 shows that they experienced a little weight loss at roughly 158°C, however, the epoxy system's main weight loss occurred between 320°C and 500°C, due to the thermal degradation of the cured epoxy





network. Jin and colleagues [32] showed that the epoxy network's thermal degradation occurred between 350° C and 500° C.



Figure 6. Results of TGA analysis (T5%, and T50%) of neat epoxy, 10 and 15 SB phr



Figure 7. DTG curves of pure epoxy, epoxy/10 phr SB and epoxy/ 15 phr SB epoxy foam

Initial degradation temperature of pure epoxy has occurred by 334° C and sustained up to 369° C. There was decomposed peak detected of SB at around 160° C, the first stage was attributed to the decomposition of SB and the dehydration of water. The results showed that, in several cases, the SB remains unreacted probably due to the very short time that the process takes place. The T_{max} of the epoxy/SB foam system (291°C) was considerably higher at the level of SB content (15 phr) as compared to the (10 phr) of epoxy/SB foam (283°C), as described in Table 1.

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TABLE 1. Weight loss Analysis by TGA of Epoxy Foam atDifferent Amounts of SB Weight Loss Temperature (°C)

-	T ₅ (°C)	T50(°C)	T _{max} (°C)
Pure epoxy	334	403	371
Epoxy/10phr SB	164	375	283
Epoxy/15 phr SB	174	385	291

6. CONCLUSION

The effects of SB as a foaming agent were examined on the cell morphology, compressive strength, and thermal characteristics of epoxy foam. The epoxy foam porosity decreased as the SB increased, as expected. The best content of SB in the epoxy foam was determined at 15 phr, where there was an increase in average pore size by increasing SB amounts, based on pore size, porosity and compression characteristics. The formation of complete bubbles by decomposed SB when the SB-foaming content was at or above the suitable limit attributed to this. This was confirmed by SEM image analysis, which showed that more pores with increased SB contents allowed the cells to come closer together, promoting the formation of additional porosity. When SB was bubbled up at optimum content into the epoxy foams of similar density. The epoxy foam's thermal stability was good with SB content when compared to pure epoxy. So the optimum proportion of SB was achieved by adding 15 phr to epoxy into the mixture. Compressive strength reduced when additional SB was used to produce epoxy foam, therefore the optimum amount was chosen. This was responsible for the increase in the epoxy foam's porosity, which correlated well with SEM morphological observations.

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