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Box–Behnken experimental design for optimization of chitosan foam materials reinforced with cellulose and zeolite

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Abstract: Foam materials produced from biopolymers stand out as a more environmentally friendly insulation material solution. This study presents a comprehensive investigation into the development and optimization of chitosan-based foam materials using a Box–Behnken design. The foams were engineered using varying proportions of chitosan (0.5–3%), cellulose (0.5–3%), and zeolite (0.5–3%), targeting their application as thermal insulators. The physical and thermal properties of the foams that were produced were affected by the type and ratios of components, with density and thermal conductivity ranging from 0.0853 to 0.1915 g cm^{-3} and 0.0324 to 0.0921 W mK^{-1}, respectively. Higher chitosan content improved insulation properties and mechanical strength whereas zeolite increments increased density and thermal conductivity. Using statistical analysis through the Box–Behnken design, we optimized the foam formulations, achieving minimum thermal conductivity and maximum compression strength at an averaged density, suggesting a strong potential for environmental sustainability applications. The recommended optimal chitosan:cellulose:zeolite composition ratio of 3:3:0.88 provides a valuable insight for tailored foam material formulation. This study shows the relationships between the composition of a composite material and its resultant properties, optimizing its preparation for industrial applicability in an environmentally conscious way within the context of insulation and construction. This investigation contributes to the field of material science by highlighting the versatility and potential of biopolymers but also aligns with the increasing need for green building materials. © 2024 The Authors. *Biofuels, Bioproducts and Biorefining* published by Society of Industrial Chemistry and John Wiley & Sons Ltd.

Key words: chitosan; insulation material; zeolite; Box–Behnken; optimization

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Introduction

The search for innovative materials has resulted in significant research with the aim of improving the properties and functionalities of materials in various industries. Foam materials, known for features such as low density, high porosity, and customizable mechanical durability, have gained prominence due to their diverse potential applications. Applications include energy absorption, drug delivery, sound absorption, filtration, tissue engineering, and thermal insulation. The properties of foam materials depend on their composition, morphology, and porosity, which can be customized by selecting different matrix and reinforcement materials and controlling the foaming process.[1] The raw materials used in foam production are synthetic polymers, metals, or ceramics. However, due to the environmental impact, cost, and limited resources of these materials, researchers have turned their attention to the production of foam materials from natural resources.[2] In recent years, the increased focus on eco-friendly and sustainable solutions and materials in research and development has become one of the top priorities for industry and the scientific community.[3–5] The use of innovative raw materials obtained from abundant natural sources such as chitosan and cellulose has the potential to reduce the environmental impacts of many industrial applications.

Chitosan is a natural polysaccharide derived from the deacetylation of chitin, which is abundant in the exoskeletons of crustaceans and insects.[6] Chitosan has many advantages as a matrix material for biodegradable foams, including biocompatibility, biodegradability, antimicrobial activity, low toxicity, and low cost. Its poor mechanical qualities, high water absorption rate, and limited thermal stability are some of its drawbacks.[7] Chitosan therefore needs to be modified or blended with other materials to enhance its performance and functionality.[8]

Cellulose, another natural polysaccharide commonly found in plants and some microorganisms, exhibits high mechanical strength, surface area, crystallinity, and good biological degradability.[9] Chitosan-based foams can have their mechanical and thermal properties enhanced by the addition of cellulose as a reinforcing ingredient.[10] In addition to using geopolymer materials to improve some properties of foam materials, one of these materials are zeolites.

Zeolites are a group comprising over 40 minerals. Among the most well-known ones are analcime, chabazite, klinoptilolite, erionite, ferrierite, heulandite, mordenite, stilbite, and phillipsite. Zeolites have a microporous structure that can exchange ions and adsorb gases. They are utilized in various applications, such as oxygen production, water purification, catalysis, and agriculture.[11] Zeolites have also been investigated for their unique properties and potential uses in energy production and the manufacture of composites.[12]

The properties of the foam material produced are influenced by the polymers used in the production process and their mixing ratios. However, measuring the effect of each parameter can be time consuming and costly, and it is crucial to have sufficient test when predicting output values. From an industrial perspective, it is desirable to determine the most suitable production conditions without raw material or product loss. Economical methods are therefore needed to achieve the desired properties of foam materials, such as compression modulus, thermal conductivity, and density.

The Box–Behnken experimental design method is a commonly used statistical approach to determine interactions and optimal regions of multiple variables.[13] This method assists in systematically changing the levels and interactions of experimental factors to identify the optimal conditions for obtaining the required material properties.[14]

Biobased foams, which either oven-dried or freeze-dried, along with commercial foams, have the intermediate-class thermal insulation properties.[15] Supercritical dried foams have equally dispersed nanosized gaps and improved heat conductivity in comparison with freeze-dried or oven-dried foams.[16] However, due to the difficulty of manufacture and the restricted production capacity, supercritical drying technology is not preferred in commercial production. Considering the production capacity, oven drying is therefore superior to supercritical drying or freeze drying.[17] In contrast with previous studies, which often focused on single components, our work delves into the combined influence of chitosan, cellulose, and zeolite in foam formulations. The utilization of a Box–Behnken design allows for a comprehensive investigation of the interactions among these components in terms of thermal, mechanical, and density properties, with a reduced number of experiments and lower costs. Aligned with the increasing demand for eco-friendly materials, this study adopts a holistic approach to environmental sustainability, emphasizing the use of readily available natural resources such as chitosan and cellulose. The inclusion of zeolite, known for its unique properties, further enhances the environmentally sustainable aspect of our approach. Optimization studies are also quite limited due to the heterogeneous structure of biobased foam materials. Zeolite, which is a geopolymer, is a material with fire resistance and, as it has a porous structure, it contributes to both fire resistance and improving the room air in the environment used.

This study aimed to produce foam materials from chitosan (0.5%, 1.75%, and 3%), cellulose (0.5%, 1.75%, and 3%), and zeolite (0.5%, 1.75%, and 3%). The effects of foam

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compositions with different chitosan, cellulose, and zeolite levels on density, compression modulus, and strength were investigated. The thermal conductivity of the foams was assessed for their potential for thermal insulation purposes. The optimal compositions of the foams were determined using the Box–Behnken design.

Materials and methods

Materials

The cellulose was purchased from Konya Paper Industry and Trade Inc. (Konya, Turkey). The density of the cellulose was 0.53 g cm^{-3}. The medium molecular weight chitosan (density: 0.2 g cm^{-3}) and citric acid (density: 1.65 g cm^{-3}) were provided by Sigma-Aldrich (Schnelldorf, Germany). Sodium dodecyl sulfate (SDS) were provided by Aromel Chemistry (Konya, Turkey). In this study, zeolite of the klinoptilolite type supplied from Rota Mining (Gordes, Manisa, Turkey), was utilized. It had a density of 2.43 g cm^{-3}, porosity of 47 m^2 g^{-1}, and the klinoptilolite content was 95%.

Methods

Box–Behnken experimental design

Design of experiments (DOE) has been widely used to optimize analytical procedures in recent years due to its benefits, such as a reduction in the number of experiments that must be performed, resulting in reduced energy and raw material consumption and significantly less laboratory effort.[18,19] Box and Wilson investigated the connections between response and explanatory variables using response surface methodology (RSM).[20] Response surface methodology is a method that combines statistical techniques to design experiments, create models, evaluate the effects of factors, and investigate the appropriate conditions to generate the desired response.[21] This approach typically combines factorial design techniques like central composite and Box–Behnken designs.[22] Box and Behnken developed the Box–Behnken design (BBD), which is a second-degree independent design under RSM.[23] It is a rotatable design class that requires three levels for each factor, such as +1, 0, and −1; +1 is the maximum, 0 is the middle, and −1 is the minimum level. The number of experiments (N) needed for the development of the Box–Behnken matrix is defined as $N=2k\,(k-1)+C0$, where (k) is the factor number and ($C0$) is the replicate number of the central point.[24,25]

The dry weight consistency of the foam mixtures, containing 0.5%, 1.75%, and 3% (w/w) of chitosan, cellulose, and zeolite, along with the experimental design using Box–Behnken Design (BBD) for the foams produced in this study, are presented in Table 1. Minitab program was used to determine the BBD and to perform the analyses.

Production process of chitosan-based foam

Chitosan dissolves in acidic environments, so distilled water creates a stock citric acid solution. The first step involved stirring chitosan at 1200 rpm for 2 h at pH 3.6 in citric acid. Different amounts of cellulose and zeolite were added, and the mixture was agitated for 10 min at 750 rpm. The mixture was stirred at 800 rpm for 10 min with the addition of 8 g L^{-1} SDS to generate bubbles. In all mixing procedures, a vortex formed. Table 1 provides information on the composition of foams.

The aqueous suspension was placed in a mold and dried at 70°C for 12 h. The process for the production of the foam is shown in Fig. 1.

Characterization of chitosan-based foam

Before measuring physical and mechanical properties, all foams were conditioned for 24 h at 20°C and 65% relative humidity. The density of the foamed material was determined according to ASTM standard C303 (2010). Compression properties were assessed using universal testing equipment (Instron 600 DX, Instron, Norwood,MA, USA) according to ASTM C165-07 (2017). The size of foams was 10 × 10 × 2 cm^3. The final strain was fixed at 25% of the original specimen height, and the compression rate was set at 8 mm min^{-1}. Thermal conductivity was measured using a heat flux (KEM QTM 500, Kyoto Electronics, Kyoto, Japan) in accordance with ASTM C518 (2021). The morphologies of the foams were studied using a JSM-7600F brand (Tokyo, Japan) scanning electron microscope (SEM). Gold was applied to the surfaces of the foams to boost conductivity. The microscope's working voltage was set at 15 kV for the examination of the microstructure images. Density, compression and thermal conductivity tests were conducted with six repetitions each.

Results and discussion

Experimental results of chitosan based foams

This study investigated the properties of foam materials with varying ratios of chitosan, cellulose, and zeolite. As shown in Table 2, the thermal conductivity and density values changed with the component types and ratio. Based on the results, the thermal conductivity and density were affected predictably by the concentration ratio of each component.

The density of the foam materials ranged from 0.0853 to 0.1915 g cm^{-3}. The lowest density was observed in the C-5

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Table 1. Composition of the foam.

Codes	Chitosan ratio (%)	Cellulose ratio (%)	Zeolite ratio (%)	Chitosan ratio (%)	Cellulose ratio (%)	Zeolite ratio (%)
C-1	–1	–1	0	0.5	0.5	1.75
C-2	1	–1	0	3	0.5	1.75
C-3	–1	1	0	0.5	3	1.75
C-4	1	1	0	3	3	1.75
C-5	–1	0	–1	0.5	1.75	0.5
C-6	1	0	–1	3	1.75	0.5
C-7	–1	0	1	0.5	1.75	3
C-8	1	0	1	3	1.75	3
C-9	0	–1	–1	1.75	0.5	0.5
C-10	0	1	–1	1.75	3	0.5
C-11	0	–1	1	1.75	0.5	3
C-12	0	1	1	1.75	3	3
C-13	0	0	0	1.75	1.75	1.75
C-14	0	0	0	1.75	1.75	1.75
C-15	0	0	0	1.75	1.75	1.75

Note: The ratios of component (%) are given by weight.

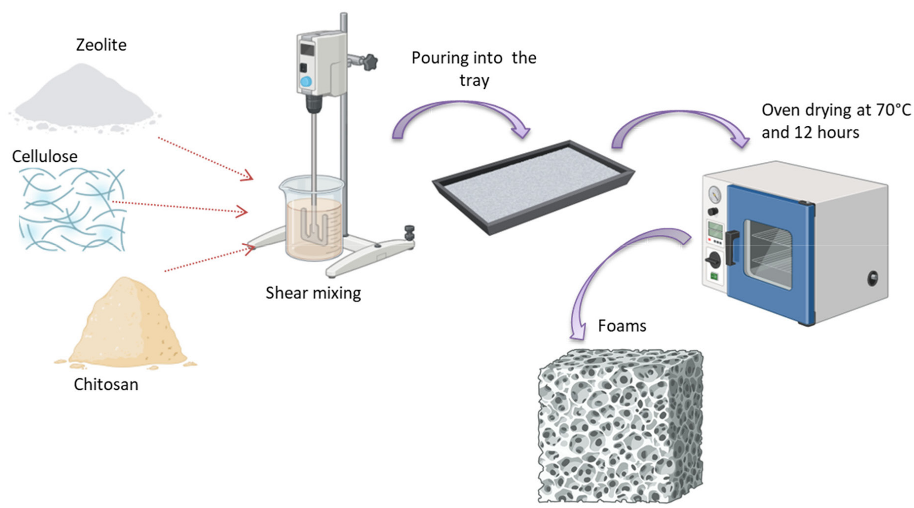

Figure 1. Chitosan-based foam production process.

combination (0.5% chitosan, 1.75% cellulose, 0.5% zeolite), whereas the highest density was found in C-12 (1.75% chitosan, 3% cellulose, 3% zeolite). The density variation can be attributed to the different composition ratios of the foams. Including a higher proportion of zeolite also had a noticeable effect on the density. These density trends may occur due to the differences in the physical properties of the constituent materials.

The aggregation of fibers during solvent removal leads to the formation of denser foams. This is due to attractive forces and capillary action between the fibers. Particle adhesion is also a contributor to the situation. The source of particles can also impact their dimensions, and using the inherent morphology of cellulose sources has advantageous effects on fiber foams.[2] The density of the foam may be diminished effectively by the selection of naturally weightless fibers, such as cellulose, which have a density of 0.53 g cm^{-3}. Biobased foams can be produced utilizing different drying methods, such as freeze drying and oven drying. The density of biobased foams manufactured with freeze drying varied

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Table 2. Thermal conductivity, mechanical properties, and density of chitosan-based foams.

Codes	Density (g cm^{-3})	Compression strength (MPa)	Compression modulus (MPa)	Thermal conductivity (W mK^{-1})
C-1	0.0968 (0.0064)	0.0218 (0.0081)	0.0901 (0.0343)	0.0336 (0.0034)
C-2	0.1337 (0.0039)	0.0280 (0.0071)	0.1150 (0.0259)	0.0518 (0.0014)
C-3	0.1082 (0.0085)	0.0274 (0.0042)	0.1097 (0.0167)	0.0361 (0.0019)
C-4	0.1376 (0.0077)	0.0557 (0.0016)	0.2226 (0.0063)	0.0529 (0.0018)
C-5	0.0853 (0.0052)	0.0197 (0.0072)	0.0790 (0.0289)	0.0324 (0.0020)
C-6	0.1183 (0.0024)	0.0356 (0.0017)	0.1424 (0.0069)	0.0439 (0.0040)
C-7	0.1109 (0.0039)	0.0108 (0.0081)	0.0432 (0.0325)	0.0328 (0.0023)
C-8	0.1530 (0.0073)	0.0398 (0.0025)	0.1590 (0.0100)	0.0618 (0.0041)
C-9	0.0973 (0.0046)	0.0189 (0.0006)	0.0757 (0.0024)	0.0439 (0.0062)
C-10	0.1090 (0.0017)	0.0247 (0.0008)	0.0988 (0.0032)	0.0416 (0.0023)
C-11	0.1291 (0.0039)	0.0277 (0.0026)	0.1108 (0.0104)	0.0350 (0.0002)
C-12	0.1915 (0.0142)	0.0383 (0.0070)	0.1530 (0.0282)	0.0921 (0.0064)
C-13	0.1142 (0.0123)	0.0286 (0.0020)	0.1143 (0.0079)	0.0364 (0.009)
C-14	0.1366 (0.0116)	0.0248 (0.0018)	0.0991 (0.0085)	0.0373 (0.0085)
C-15	0.1166 (0.0125)	0.0276 (0.0016)	0.1105 (0.0083)	0.0381 (0.0094)

Note: Standard deviations indicated in parentheses.

between 0.006 and 0.21 g cm^{-3}.[2,26,27] On the other hand, foams made from biobased materials and produced by the oven-drying process have densities that range from 0.0806 to 0.49 g cm^{-3}.[10,28,29] In this study, the densities of chitosan-based foam varied between 0.0853 and 0.1915 g cm^{-3}, similar to the density range of biobased foams manufactured using freeze-drying and oven-drying methods. The density range of the chitosan foam is also similar to that of expanded polystyrene foam (EPS), which has densities ranging from 0.002 to 0.64 g cm^{-3}.[30]

The thermal conductivity values of the foams changed between 0.0324 and 0.0921 W mK^{-1}. C-12, with 1.75% chitosan, 3% cellulose, and 3% zeolite demonstrated the highest thermal conductivity, whereas C-5, with 0.5% chitosan, 1.75% cellulose, and 0.5% zeolite, exhibited the lowest thermal conductivity. The thermal conductivity of the materials is significantly affected by the composition and distribution of zeolite, which has intrinsic thermal properties. The thermal conductivity values of xanthan gum,[31] starch,[32] zinc borate,[1] and chitosan[33] foams were detected between 0.023 to 0.082 W mK^{-1}. Based on a comparison of previous studies' thermal conductivity, chitosan-based foam appears to be a suitable insulation solution. The foam's porous microstructures were found to have favorable thermal insulation properties.[34] The gaps inside the foam structure are filled by air, which has low heat conductivity. The layered structure formed in foam materials restricts thermal conductivity due to the formation of voids in the pore walls.[35] The augmentation of solid material – another crucial factor – has also resulted in a noteworthy rise in the thermal conductivity of the foams. As the foam becomes denser, the thermal conductivity of the solid components also begins to increase rapidly.[36]

The compression strength of foams ranged from 0.0108 to 0.0557 MPa. The highest compression strength was observed in C-4 (3% chitosan, 3% cellulose, 1.75% zeolite), and the lowest was observed in C-7 (0.5% chitosan, 1.75% cellulose, 3% zeolite). As the chitosan proportion increased, the compression strength improved. The highest compression strength was observed for C-4, which had a chitosan proportion of 3%. Chitosan plays a vital role in enhancing the mechanical properties of the foams. The addition of zeolite also positively influenced the compression strength, which is consistent with its higher density. The compression modulus of foams varied from 0.0432 to 0.2226 MPa. C-4, with the highest chitosan and cellulose content, exhibited the highest compression modulus, whereas C-7, with high zeolite content, showed the lowest. The type and proportion of the components influence the compression properties of the foam materials. The compression modulus, indicating the strength of materials to deformation, displayed similar trends to the compression strength. Foams with higher proportions of chitosan exhibited greater resistance to deformation.

Foam density was found to have a significant effect on compression strength and modulus. The strong correlation between strength and modulus with density is due to the interconnected structure of the foam, which consists of cellulose fibers, chemical crosslinking, and fiber-to-fiber

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contact points.[37] The compression strength of commonly used foams, including polyurethane, extruded polystyrene (XPS) and expanded polystyrene (EPS), ranged from 0.025 to 0.710 MPa.[38] Murmu[39] attributed cellulose reinforcement to increased interfacial interactions due to polar and hydrogen bonding between the reinforcing hydroxy groups and the matrix. Furthermore, the hydroxyl groups and amino present in cellulose and chitosan are responsible for forming intermolecular hydrogen and covalent bonds, resulting in enhanced compression properties.[40] Citric acid contains abundant carboxyl groups, which can acylate with the flexible molecular chains of protonated chitosan by reaction between $-NH_3^+$ and $-COOH$ to form a crosslinked polymer.[41] The crosslinking of chitosan with citric acid results in an augmentation of the viscoelastic behavior of materials, leading to improved mechanical properties.[42] Consequently, citric acid can be employed to dissolve chitosan and facilitate crosslinking with both cellulose and chitosan.[43] Cellulose and chitosan-based or reinforced foams were investigated in numerous studies. Compression strength and modulus of guar gum, chitosan, starch, and cellulose-based foams were found between 0.01 to 3.3 MPa[44–46] and 0.016 to 4.5 MPa,[47–49] respectively.

The mechanical resistance properties of materials incorporating inorganic fillers such as zinc borate, zeolite, and calcium carbonate were influenced either positively or negatively depending on the usage ratio of these fillers.[50,51] Zeolite is often used in significant quantities during material production but it tends to clump together within the polymer matrix, causing a decline in the compression properties of materials. This is typically attributed to insufficient dispersion of inorganic fillers in the polymer matrix, which results in an accumulation of aggregate that acts as stress points and causes material degradation.[52,53] On the other hand, chitosan foams have a stronger structure because of the covalent connections between the negatively charged cellulose fiber and the positively charged chitosan. Furthermore, interpenetrating polymer networks can be formed by hydrogen bonding and physical entanglements between cellulose, zeolite, and chitosan.[54]

These were the SEM images of the samples C-4 with the highest mechanical properties, C-5 with the lowest thermal conductivity and density, C-12 with the highest density and thermal conductivity, and C-15 with average values, as shown in Fig. 2.

The porous structure of chitosan-based foam materials is shown in Fig. 2. The cell wall of the C-4 with the highest chitosan and cellulose ratio is shown with green arrows. As Fig. 2(a) shows, the compression properties improved with increasing chitosan content, which gave the shape and basic qualities of the foam material, was closely related to having a thicker cell wall.[55] On the other hand, the foam material with the lowest density (C-5) had more and wider pores than the other samples, as Fig. 2(b) shows. The density, thermal conductivity, and mechanical characteristics decreased in direct proportion to the size and abundance of the porous structure.[47] The internal structure of the foam material with high zeolite content was shown in Fig. 2(c). The zeolite clusters on cellulose and chitosan in the foam material are shown in blue circles. This clustering reduced bonding between cells and caused a decrease in mechanical properties. Similar studies have shown that clustering occurs in foam materials with excessive filler addition.[10,56,57] The cell wall formed by chitosan surrounding cellulose is shown in Fig. 2(d). The foams clearly display a network of interconnected pores. The foams have an irregular porous layered structure (Fig. 2(a–d)). The structures of the foams vary depending on the amount of zeolite and chitosan. Chitosan aggregates large bulks at higher concentrations, which limits the mobility of the masses and influences their rearrangement. Bubble formation is also hindered by an increase in solution viscosity as the amount of chitosan is increased. When the chitosan concentration was 3%, the lamellar texture was replaced by dense texture (Fig. 2(a)) because of the increasing solution viscosity and decreasing mass mobility of the precursor solutions.[2] The addition of zeolites to the precursor solution resulted in the encapsulation of the zeolites in chitosan due to the strong hydrogen bond. A few struts are also seen to bridge the gaps among the zeolite particles (Fig. 2(c)). Hydroxyl groups in the matrix are known to form hydrogen bonds with inorganic particles.[58] The cellulose reduced the interpore gap. The morphological structures of nanofibril cellulose and starch foam materials had uniform pore shapes,[59] but the morphological structure of chitosan foams was shown to be dense and irregular. The cellulose particles were macro-sized. This disturbed the interpore spaces and structure. Similar dense and irregular structures were obtained with cellulose-based foams.[44] In previous studies, the morphological structure of the foams had a honeycomb shape.[59,60] Nevertheless, this study noted irregularities in the morphological structures of the foams. This is due to the oven drying technique and the macrosized cellulose that disturbs the cell structures and interstices. Previous studies found that foams produced from biopolymer derivatives such as chitosan,[61] starch,[62] and cellulose[63] had irregular and open cell pore structures.

Analysis of variance

The requirements for the use of analysis of variance (ANOVA) were met, enabling the use of parametric tests and the implementation of analyses. A Box–Cox transformation

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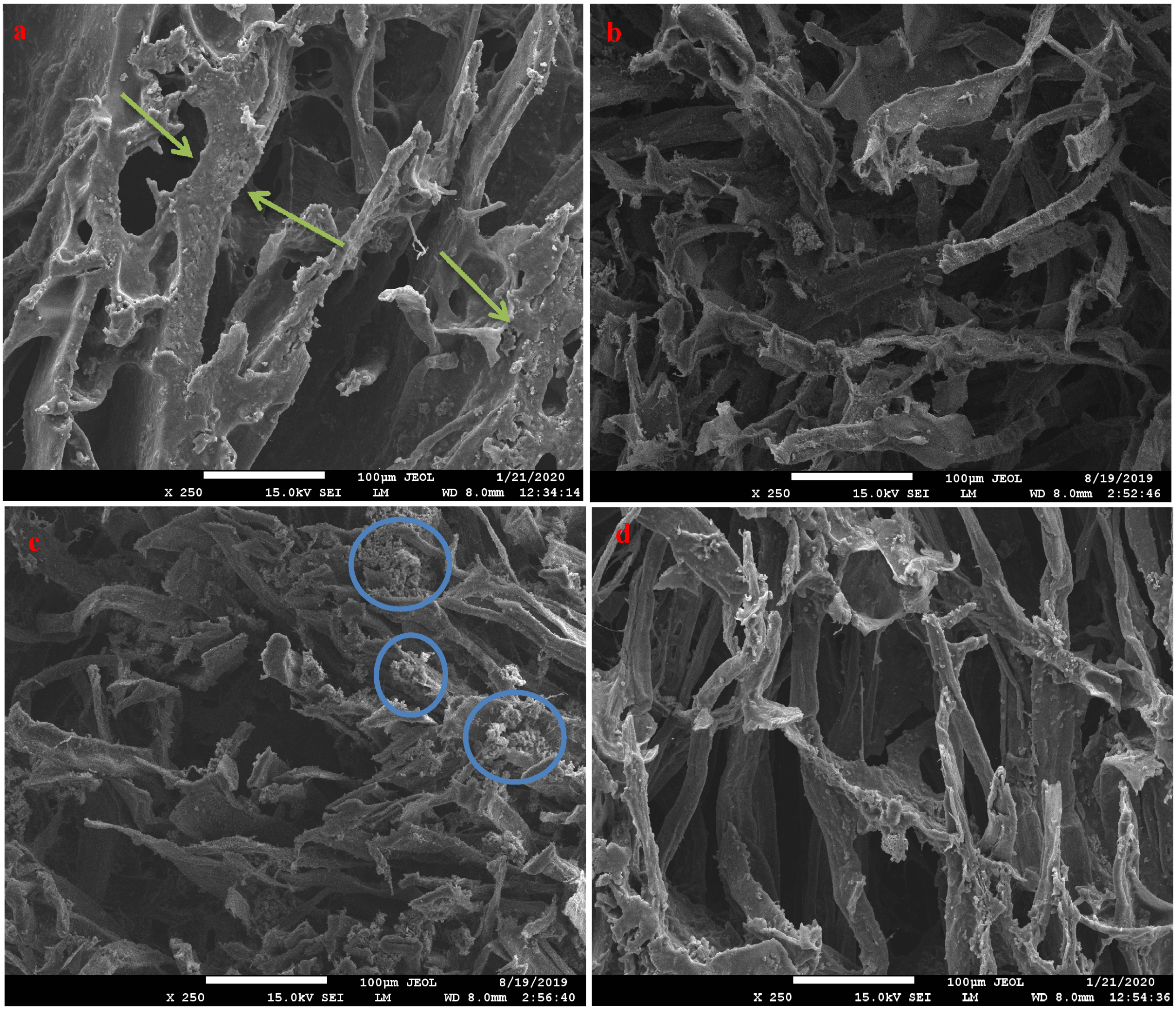

Figure 2. Scanning electron microscopy image of chitosan based foams (a. C-4, b. C-5 c. C-12 and d. C-15).

was performed on the skewed data to enable an assumption of normality to be made regarding thermal conductivity data. Table 3 provides the results of the variance analysis for the linear relationships between the thermal conductivity, compression strength, compression modulus, and density of the foam material used in production, as well as their pairwise interactions. When ANOVA results were examined, no generally significant results were obtained between pairwise interactions and squares of variables at a 5% significance level. When the effects of linear variables were investigated, it was observed that using chitosan and cellulose significantly influenced thermal conductivity, compression strength, and compression modulus. In contrast, the use of chitosan and zeolite had significant effects on density. Chitosan, functioning as the matrix material shaping the fundamental structure, was observed, in SEM images, to enhance density by forming thicker cell walls. In contrast, the impact of zeolite on density was more pronounced in conjunction with chitosan than with cellulose, owing to the significantly higher density of zeolite compared to the other two biopolymers. Moreover, the integration of zeolite particles into the foam may fill the spaces between foam cells (Fig. 2(c)), resulting in a denser overall structure.

The combined use of chitosan and zeolite demonstrated a synergistic effect on foam density, as both polymers contribute to an increased mass per unit volume. Conversely, the inclusion of cellulose tended to counteract this effect by reducing the mass per unit volume. Consequently, the utilization of chitosan and zeolite exerted a more substantial influence on foam density than cellulose. Previous studies

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Table 3. Analysis of variance results.

Source	Density					Thermal conductivity				
	df	SS	MS	*F*	*P*	df	SS	MS	*F*	*P*
Model	9	0.008500	0.000944	4.26	0.063	9	862 641	95 849	12.03	0.007
Linear	3	0.007309	0.002436	10.99	0.012	3	611 540	203 847	25.58	0.002
A	1	0.002499	0.002499	11.27	0.020	1	512 308	512 308	64.29	0.000
B	1	0.000999	0.000999	4.50	0.087	1	74 739	74 739	9.38	0.028
C	1	0.003811	0.003811	17.18	0.009	1	24 493	24 493	3.07	0.140
Square	3	0.000514	0.000171	0.77	0.557	3	91 347	30 449	3.82	0.092
A*A	1	0.000307	0.000307	1.39	0.292	1	1003	1003	0.13	0.737
B*B	1	0.000121	0.000121	0.55	0.493	1	73 918	73 918	9.28	0.029
C*C	1	0.000046	0.000046	0.21	0.668	1	18 912	18 912	2.37	0.184
Two-way interaction	3	0.000677	0.000226	1.02	0.458	3	159 755	53 252	6.68	0.034
A*B	1	0.000014	0.000014	0.06	0.811	1	2657	2657	0.33	0.589
A*C	1	0.000021	0.000021	0.09	0.772	1	13 684	13 684	1.72	0.247
B*C	1	0.000643	0.000643	2.90	0.149	1	143 413	143 413	18.00	0.008
Error	5	0.001109	0.000222	–	–	5	39 845	7969	–	–
Lack-of-fit	3	0.000806	0.000269	1.78	0.380	3	37 670	12 557	11.55	0.081
Pure error	2	0.000303	0.000151	–	–	2	2174	1087	–	–
Total	14	0.009609	–	–	–	14	902 486		–	–
	Compression strength					Compression modulus				
Source	df	SS	MS	*F*	*P*	df	SS	MS	*F*	*P*
Model	9	0.001424	0.000158	5.32	0.040	9	0.022616	0.002513	5.49	0.038
Linear	3	0.001136	0.000379	12.73	0.009	3	0.017807	0.005936	12.96	0.009
A	1	0.000788	0.000788	26.48	0.004	1	0.012561	0.012561	27.43	0.003
B	1	0.000309	0.000309	10.38	0.023	1	0.004632	0.004632	10.11	0.025
C	1	0.000039	0.000039	1.32	0.303	1	0.000614	0.000614	1.34	0.299
Square	3	0.000117	0.000039	1.31	0.368	3	0.002095	0.000698	1.52	0.317
A*A	1	0.000026	0.000026	0.87	0.393	1	0.000476	0.000476	1.04	0.355
B*B	1	0.000047	0.000047	1.59	0.264	1	0.000834	0.000834	1.82	0.235
C*C	1	0.000037	0.000037	1.25	0.314	1	0.000665	0.000665	1.45	0.282
Two-way interaction	3	0.000171	0.000057	1.91	0.246	3	0.002714	0.000905	1.98	0.236
A*B	1	0.000122	0.000122	4.10	0.099	1	0.001936	0.001936	4.23	0.095
A*C	1	0.000043	0.000043	1.44	0.284	1	0.000686	0.000686	1.50	0.275
B*C	1	0.000006	0.000006	0.19	0.678	1	0.000091	0.000091	0.20	0.674
Error	5	0.000149	0.000030	–	–	5	0.002290	0.000458	–	–
Lack-of-fit	3	0.000141	0.000047	12.12	0.077	3	0.002165	0.000722	11.53	0.081
Pure error	2	0.000008	0.000004	–	–	2	0.000125	0.000063	–	–
Total	14	0.001573	–	–	–	14	0.024906	–	–	–

Note: A: chitosan, B: cellulose, C: zeolite, SS: sum of squares, df: degrees of freedom, MS: mean squares.

found similar results.[47,50] In general, a high coefficient of determination (R^2) of over 90% was obtained among all variables, and a good fit was determined between the experimental data and the data estimated by the model.

Figure 3 presents a graph that reveals the effects of chitosan, cellulose, and zeolite usage on the thermal conductivity, compression strength, compression modulus, and density of the foam material. It can be seen that high chitosan

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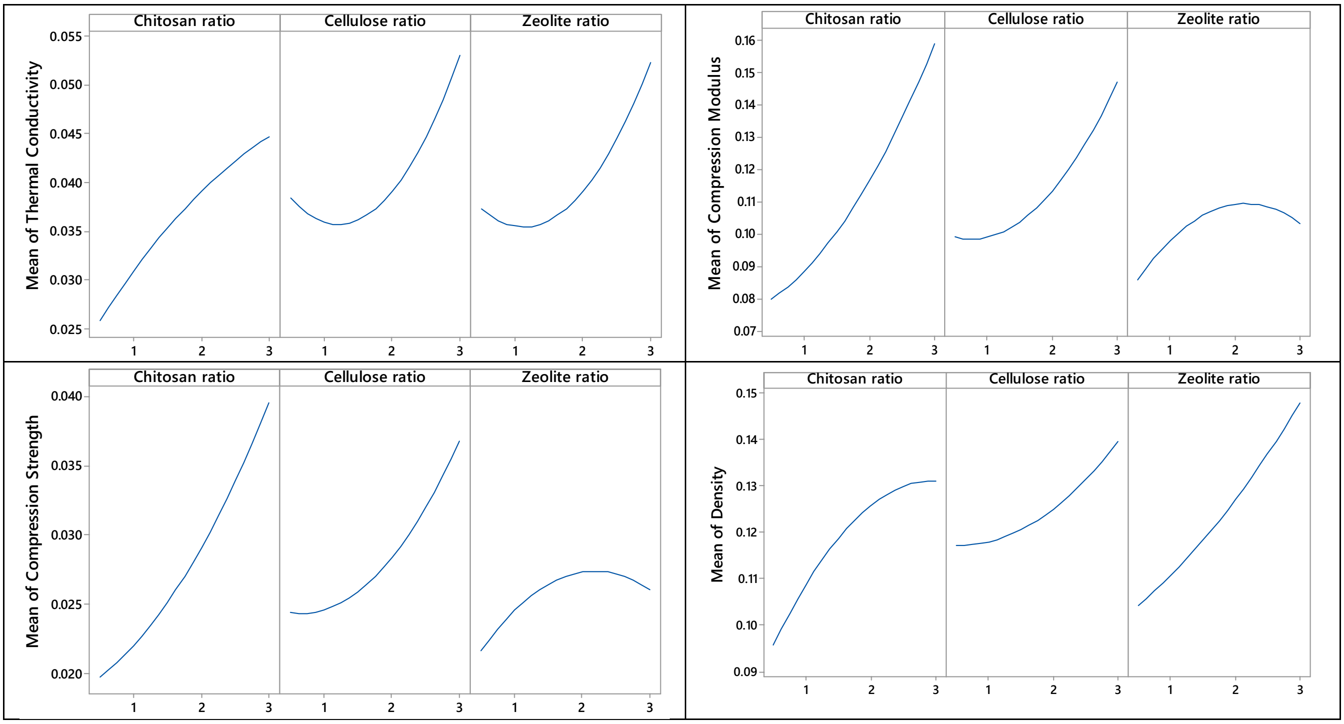

Figure 3. Surface plot and contour plot graphs of the dual effects on density and thermal conductivity.

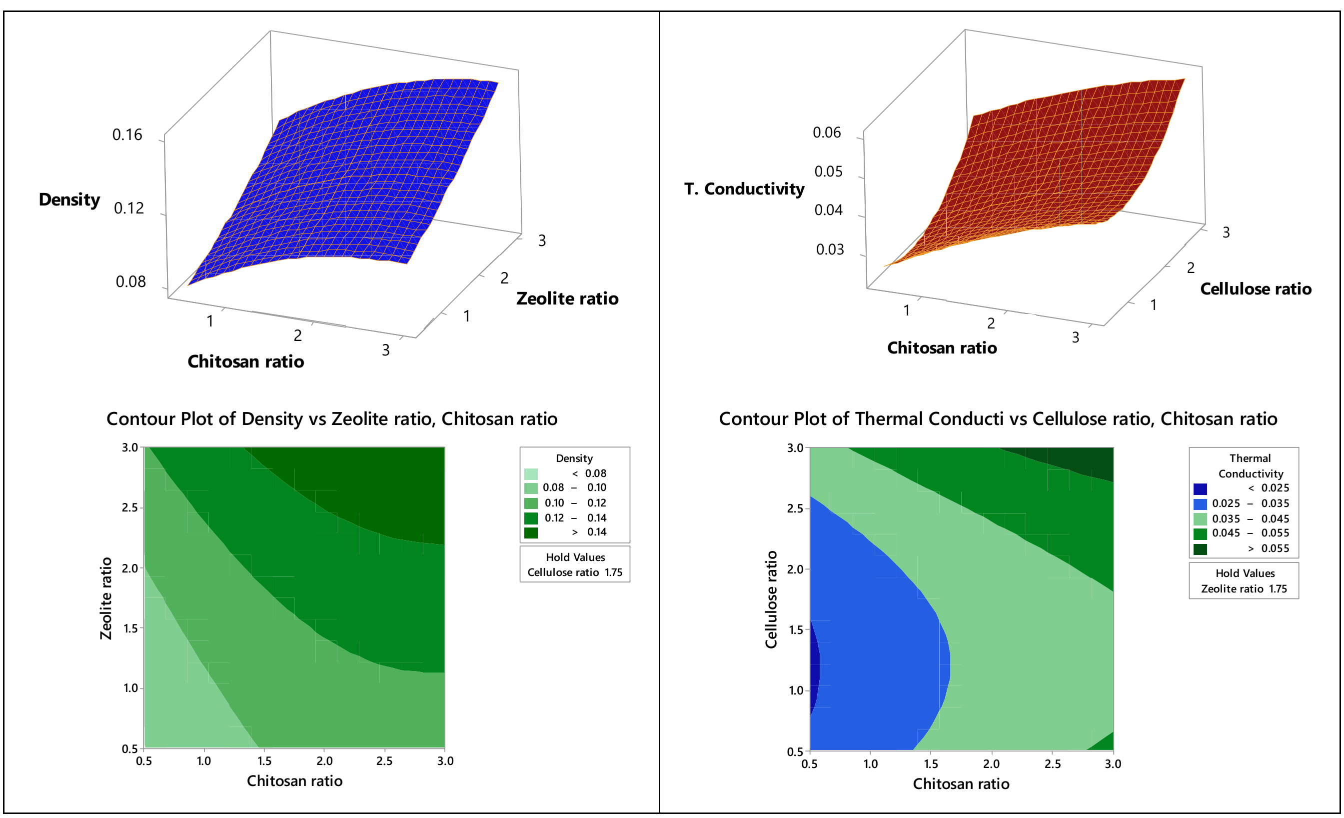

Figure 4. Surface plot and contour plot graphics of density and thermal conductivity values.

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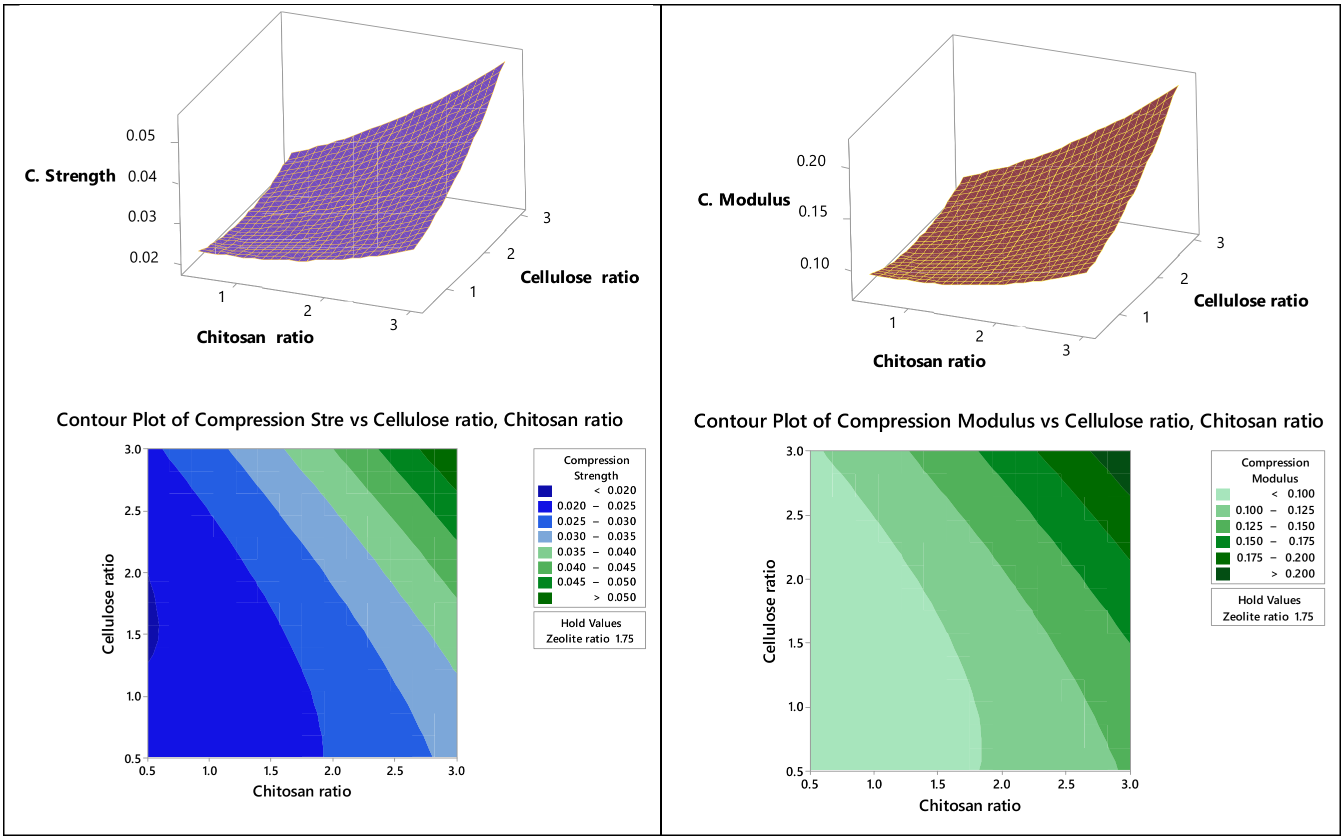

Figure 5. Surface plot and contour plot graphics of compression strength and modulus values.

usage positively affects all properties. The use of cellulose and zeolite initially reduces thermal conductivity but then increases it. Cellulose also has a positive effect on all other properties. The use of zeolite significantly increases density.

Figure 4 presents surface and contour plot graphics showing the binary effects of significant variables obtained from the ANOVA analysis of density and thermal conductivity. The use of chitosan and zeolite has a positive impact on the density of the foam materials that are produced. Optimal density values can be achieved using approximately 2–3% zeolite and 1.5–3% chitosan. Use of high amounts of chitosan and cellulose increase thermal conductivity. Desired minimum thermal conductivity values can be achieved by using 1–1.5% cellulose and 0.5% chitosan.

Figure 5 presents surface and contour plot graphics showing the binary effects of significant variables obtained from ANOVA analysis on compression strength and modulus. Both compression strength and modulus values exhibit similar properties to the use of chitosan and cellulose. In general, the mechanical properties of the foam material improve as the use of chitosan and cellulose increases. The best compression strength and modulus values are obtained with 3% chitosan and cellulose use.

Figure 6 provides the optimal conditions for the variables used in the foam materials. It was desired that foams possess the minimum thermal conductivity and maximum compression properties. The mechanical properties of high-density foam materials are favorable and desirable; however, their high density also increases thermal conductivity, which is an undesirable characteristic for insulation materials. Therefore, average density value of 0.122 in our measurements was chosen to strike a balance between these conflicting requirements. The optimum chitosan:cellulose:zeolite ratio, which met all of the requirements, was 3:3:0.88.

Conclusion

In this study, foam materials with different ratios of chitosan, cellulose, and zeolite were prepared, characterized, and optimized. The results showed that the composition and distribution of the components had significant effects on the physical, thermal, and mechanical properties of the foams. The foams with higher chitosan content exhibited lower density, lower thermal conductivity, and higher compression strength and modulus. The addition of zeolite also increased the density and thermal conductivity of the foams but

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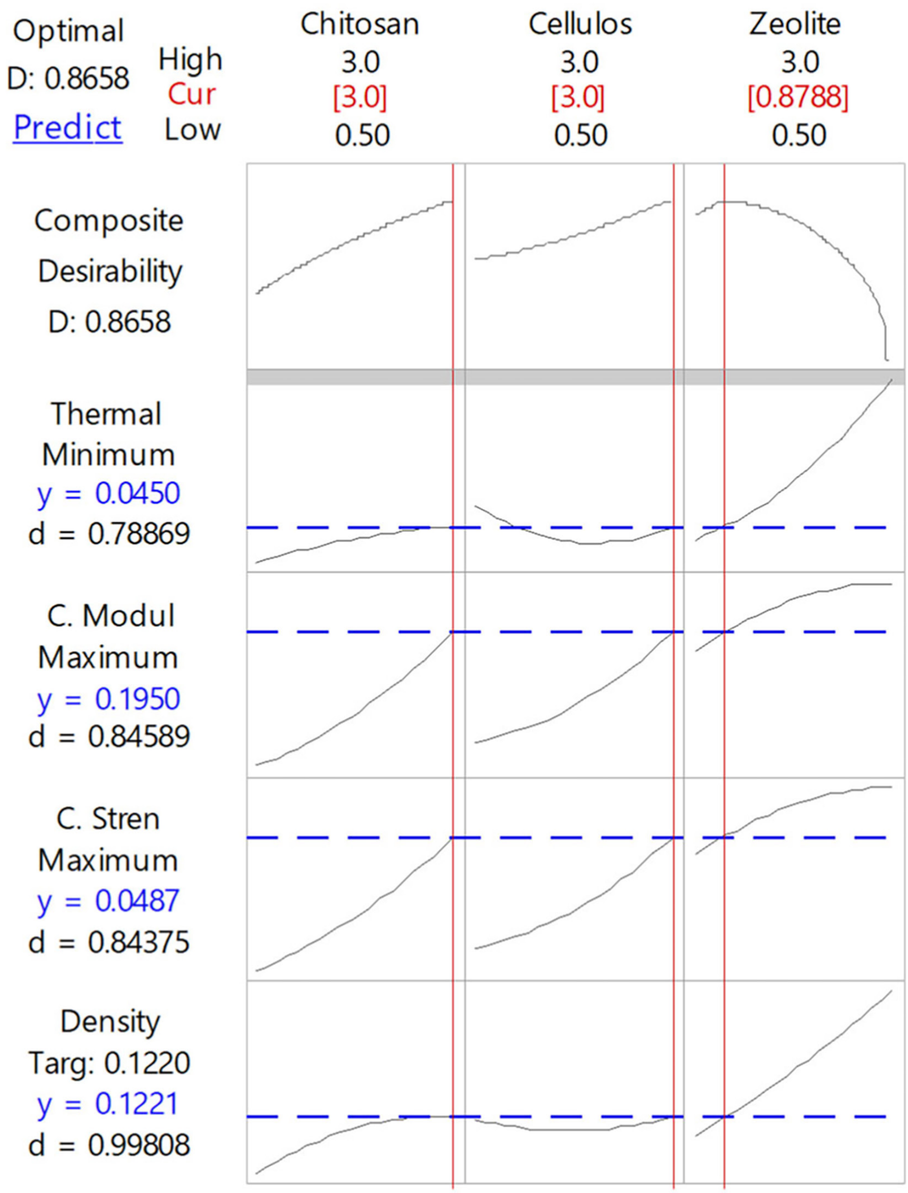

Figure 6. Optimum production parameters for chitosan-based foams.

decreased their compression strength and modulus. The foams had porous and layered structures, contributing to their thermal insulation and mechanical resistance properties. The foams also had comparable or superior properties to other biobased and commonly used foams, such as EPS and XPS.

On the other hand, the experimental results provide valuable insights for optimizing foam material composition with Box–Behnken design. The recommended chitosan:cellulose:zeolite ratio of 3:3:0.88 offers optimal conditions for achieving minimum thermal conductivity, maximum compression strength, and an average density of 0.122. The study highlights the potential of chitosan-based foams as a promising insulation solution, supported by a robust statistical foundation and a thorough understanding of the material-property relationships. Finally, chitosan-based foam is proposed as a viable insulation solution that not only meets performance criteria but also complies with environmental sustainability goals.

Author contributions

Rıfat Kurt and Halime Ergun: Conceptualization, methodology, formal and statistical analysis, and original draft. Mehmet Emin Ergun: Material preparation and testing,

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writing and editing of original draft. Abdullah Istek: Data curation, writing and editing of original draft.

Conflict of interest

The authors declare no conflict of interest.

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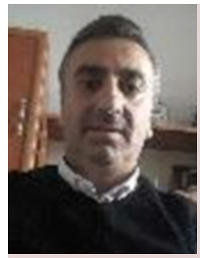

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