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Optimization of Liquefaction Parameters of Cotton Burrs (*Gossypium hirsutum* L.) for Polyurethane-Type Isolation Foams

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Abstract

Aim of study: The use of composites obtained from wood and similar lignocellulosic plants has increased all over the world. Because of the rapid depletion of forest natural resources, the rational use of declining wood raw materials and to evaluate environmental alternatives has gained importance. For this purpose, many alternative lignocellulosic raw materials are used as heat insulation material by producing foams.

Material and method: This research compared the properties of liquefied using cotton burr after liquefaction under different acid concentrations, PEG 400-Glycerin/Cotton burr ratios, pore sizes, and PEG 400/Glycerin ratios. The cotton burrs were put in a reaction chamber at 160 °C for 2 h with the PEG 400-Glycerin/cotton burr and sulfuric acid in a glass balloon.

Main results: The maximum yield in the liquefaction of cotton burrs was found in the polyethylene glycol/glycerin ratio of 1:1. It has been found that the amount of unliquefied cotton burrs decreased with increasing of the acid concentration, reaction temperature and time.

Research highlights: Consequently, the liquefaction of cotton burrs could be seamlessly used in the production of polyurethane-type foam.

Keyword: Liquefaction, Cotton Burr, Polyurethane, Foam, Wood

Poliüretan Tipi İzolasyon Köpükleri İçin Pamuk Kozalarının (*Gossypium hirsutum* L.) Sıvılaştırma Parametrelerinin Optimizasyonu

Öz

Çalışmanın amacı: Odun ve benzeri lignoselülozik bitkilerden elde edilen kompozitlerin kullanımı tüm Dünya'da artmıştır. Orman doğal kaynaklarının hızla tükenmesi nedeniyle, azalan odun hammaddelerinin rasyonel kullanımı ve çevresel alternatiflerin değerlendirilmesi önem kazanmıştır. Bu amaçla, birçok alternatif lignoselülozik hammaddeden köpük üretilerek ısı yalıtım malzemesi olarak kullanılır.

Materyal ve yöntem: Bu araştırmada, pamuk kozaları farklı asit konsantrasyonları, PEG 400-Gliserin/Pamuk koza oranları, gözenek boyutları ve PEG 400/Gliserin oranlarında sıvılaştırılarak sıvılaşmanın özellikleri karşılaştırılmıştır. Pamuk kozaları, bir cam balon içerisinde PEG 400-Gliserin/pamuk kozası ve sülfürik asit ile 2 saat boyunca 160 °C sıcaklıkta bir reaksiyona tabi tutulmuştur.

Temel sonuçlar: Pamuk kozalarının sıvılaştırılmasındaki en iyi sonuç, polietilen glikol/gliserin (1: 1 oranı) ile belirlenmiştir. Asit konsantrasyonunun, sıcaklığın ve zamanın arttırılması ile sıvılaştırılmamış pamuk kozalarının miktarında bir düşüş olduğu tespit edilmiştir.

Araştırma vurguları: Sonuç olarak, pamuk kozalarının sıvılaştırılması, poliüretan tipi köpük üretiminde kullanılabilir olmasının uygun olduğu kanaatine varılmıştır.

Anahtar Kelimeler: Sıvılaştırma, Pamuk Kozası, Poliüretan, Köpük, Odun

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Introduction

Non-renewable fossil fuel resources are quickly decreasing, and the effect of costly prices has created the fast increasing need for newer cheaper energy resources. Turkey has obtained energy that is more expensive from its neighbor countries. The country's problem of staying ahead of industrialization and development needs to be solved immediately (Rowell et al., 1992; Hasselgren, 1998).

The chemicals used in synthetic foam have an adverse effect on the environment because of an oil residue that increases the amount of CO_2 released in the air and thus accelerates global warming. Alternatively, by using annual plants, these obstacles are removed, which may reduce a major environmental effects.

Wood or its derivatives has been one of the foremost energy sources throughout human history (Brolund & Lundmark, 2014). It is a natural renewable resource. It can be used in a wide range of ways. In this days, it is more widely used in industry. In developing countries, wood constitute major energy source but, in developed countries, wood processing is rational and extensive (Stolarski et al., 2013). The shortage of wood, its increasing price and the growing competition for this material between the pulp and paper industry, the energy sector using biomass and manufacturers of boards (Dziurka & Mirski, 2013).

The use of composites made of wood and other lignocellulosic plants has been increasing globally due to forest resources being rapidly depleted. Evaluations of environmentally alternative materials as well as studies of declining wood raw materials are very important. For this purpose, foams that are produced from many alternative raw materials are used as thermal insulation materials (Alma et al., 2003; Alma, 2005).

Rice, wheat straw, soybean, kraft lignin, solvolysis lignin, bagasse, molasses, wood flour, unmodified bark, modified corn starch, coffee powder, used and unused wood, starch, modified starch, bark, grain, used paper, cotton, and wheat stalks have been used to produce different types of polyurethane foams through a liquefaction process (Timothy & Glasser, 1984; Shiraishi et al., 1985; Hirose et al., 1989; Kennedy et al., 1993; Shiraishi et al., 1993; Meikleham & Pizzi, 1994; Alma et al., 1995; Alma, 1996a, Alma et al., 1996b, Alma et al., 1996c; Ge & Sakai, 1996; Alma, 1997; Alfani et al., 1998; Alma et al., 1998; Alma & Shiraishi, 1998; Chian & Gan 1998; Ge & Sakai 1998; Alma & Kelley, 2000; Ge et al., 2000; Alma et al., 2002; Alma et al., 2003; Cuningham et al., 2003; Alma, 2005; Banik & Sain, 2008; Chow et al., 2008).

The exploitation of lignocellulosic biomass has attracted much attention due to the abundant reserves and renewable nature of the raw materials. Lignocellulosic biomass can be utilized to manufacture polymer materials. As lignocellulosic biomass generally presents fall processing performances. It is occasionally turned into substances that can be treated before use. A lot of conversion techniques have been proposed in the past few decades. Liquefaction is one of the efficient and useful techniques. Various liquefaction ways have been improved to complete the conversion. The liquefaction products having low molecular weight have been utilized as chemicals or fuels. The liquefaction products possessing active functional groups and moderate molecular weight can be utilized to manufacture foams, plastics, adhesives and films (Chen & Lu, 2009).

In this study, cotton burrs were liquefied with polyethylene glycol and glycerin at different acid concentrations, PEG 400-glycerin/cotton burr ratios, pore sizes, and PEG 400/glycerin ratios to determine their effectiveness for use with polyurethane foams.

The novelty of this study is that polyethylene glycol/glycerin was chosen as liquefying agent since it is more environmentally friendly and economical polyol and has also a higher rate of liquefaction. Moreover, cotton burrs were preferred due to its widespread use.

Materials and Methods

Materials

Cotton burr (*Gossypium hirsutum* L.) powders (60 mesh) were used as a primary

natural polyol in the liquefaction of polyurethane-type foams (Figure 1).

Figure 1. Cotton burr and its powder

Methods

The cotton burrs were kindly supplied by Kahramanmaraş Agricultural Research Institute, Turkey. Reagent grade polyethylene glycol (PEG 400), glycerin, sulfuric acid, and 1,4-dioxan (solvent) were donated by Ispol Ltd., (İzmir, Turkey) and used without any further purification.

The liquefied cotton burr powder polyol that was used for preparing the liquefied rigid polyurethane foams (RPUFs) was obtained using PEG 400/glycerin as a liquefaction solvent for 2 h at 160 °C, using different amount catalyst concentrations, PEG 400-glycerin/cotton burr ratios, pore sizes, and PEG 400/glycerin ratios. Three measurements were made for each treatment, and the averages were recorded (Table 1-4).

Table 1. Reaction conditions and components with varied acid (H_2SO_4) concentrations

Number	T[2] (°C)	t[3] (h)	P-G/CB[4]	H_2SO_4[5] (%)	P-G[6] (g)	PS[7] (mesh)
CB[1]$_1$	160	2	3/1	5	4/1	60
CB_2	160	2	3/1	7	4/1	60
CB_3	160	2	3/1	9	4/1	60

Notes: [1]CB: Cotton Burr, [2]T: Temperature, [3]t: Time, [4]P-G/CB: PEG 400-Glycerin/Cotton Burr Ratio, [5]H_2SO_4: Quantity of PEG 400-Glycerin Devoted to % Weight, [6]P-G: PEG 400-Glycerin Ratio, and [7]PS: Pore Size

Table 2. Reaction conditions and components with varied peg 400-glycerin/cotton burr ratios

Number	T[2] (°C)	t[3] (h)	P-G/CB[4]	H_2SO_4[5] (%)	P-G[6] (g)	PS[7] (mesh)
CB[1]$_4$	160	2	3/1	9	4/1	60
CB_5	160	2	3/2	9	4/1	60
CB_6	160	2	3/3	9	4/1	60

Notes: [1]CB: Cotton Burr, [2]T: Temperature, [3]t: Time, [4]P-G/CB: PEG 400-Glycerin/Cotton Burr Ratio, [5]H_2SO_4: Quantity of PEG 400-Glycerin Devoted to % Weight, [6]P-G: PEG 400-Glycerin Ratio, and [7]PS: Pore Size

Table 3. Reaction conditions and components with varied pore sizes

Number	T[2] (°C)	t[3] (h)	P-G/CB[4]	H_2SO_4[5] (%)	P-G[6] (g)	PS[7] (mesh)
CB[1]$_7$	160	2	3/1	9	4/1	40
CB_8	160	2	3/1	9	4/1	60
CB_9	160	2	3/1	9	4/1	80
CB_{10}	160	2	3/1	9	4/1	100

Notes: [1]CB: Cotton Burr, [2]T: Temperature, [3]t: Time, [4]P-G/CB: PEG 400-Glycerin/Cotton Burr Ratio, [5]H_2SO_4: Quantity of PEG 400-Glycerin Devoted to % Weight, [6]P-G: PEG 400-Glycerin Ratio, [7]PS: Pore Size

Table 4. Reaction conditions and components with varied peg 400/glycerin ratios

Number	T[2] (°C)	t[3] (h)	P-G/CB[4]	H_2SO_4[5] (%)	P-G[6] (g)	PS[7] (mesh)
CB[1]$_{11}$	160	2	3/1	9	1/1	60
CB_{12}	160	2	3/1	9	4/1	60
CB_{13}	160	2	3/1	9	7/1	60
CB_{14}	160	2	3/1	9	10/1	60

Notes: [1]CB: Cotton Burr, [2]T: Temperature, [3]t: Time, [4]P-G/CB: PEG 400-Glycerin/Cotton Burr Ratio, [5]H_2SO_4: Quantity of PEG 400-Glycerin Devoted to % Weight, [6]P-G: PEG 400-Glycerin Ratio, and [7]PS: Pore Size

The polyethylene glycol, glycerin, and sulfuric acid were added into a three-necked round bottom glass equipped with a reflux condenser and a stirrer (Figure 2). The cotton burr was then added into the bottom glass, which was preheated to the desired temperature. The liquefaction was conducted under constant stirring and refluxing at 160 °C. After a defined duration, the reactor was rapidly cooled to room temperature.

Figure 2. Process diagram in the liquefaction of biomass.

The reaction was concentrated via thin film evaporation at 60 °C to remove the binary solvent (dioxane/water: 4/1) and then passed through a glass-fiber filter paper. The amounts of unliquefied biomass were determined. The dioxane-water mixture was then added to the liquefied sample to determine the percent of residue, or soluble contents, remaining in the dioxane (Alma et al., 2002).

To determine the percent of the dioxane-insoluble parts (i.e., the unliquefied amount of cotton burr) of the mixtures that were obtained at the end of the wood liquefaction process, they were diluted with a 1,4-dioxane mixture and then filtered as described above. The dioxane-insoluble parts (DIP) were quantified by Eq. 1,

$$DIP = \frac{w_R}{w_S} x100 \quad (1)$$

where DIP is the dioxane-insoluble parts (%), w_R is the weight of the residue (g) and w_S is the weight of the wood sample (g) (Kurimoto et al., 1999; Yan et al., 2008).

The different acid concentrations, PEG 400-glycerin/cotton burr ratios, pore sizes, and the PEG 400/glycerin ratios of liquefaction in cotton burrs of the samples were measured in triplicate and used to conduct an analysis of variance, which employed randomized block factorial experimental design using SAS software (Cary, NC, USA).

Finally, a multiple correlation analysis was carried out to investigate the relationship between the groups studied. The values in the range of 0.50 to 0.75 were identified as moderately correlated, and those in the range of 0.75 to 1.00 were identified as having a high correlation in the multiple correlation analysis.

Results and Discussion

The results of the liquefaction of cotton burrs by the type of acid concentration, PEG 400-glycerin/cotton burr ratio, pore sizes, and PEG 400/glycerin ratio are presented in Tab. 5. The differences in the liquefaction of cotton burrs were significant at a threshold of 1% for the acid concentration, PEG 400-glycerin/cotton burr ratio, and PEG 400/glycerin ratio, and at a threshold of 5% for the type of pore size in the conducted analysis of variance (Table 5).

Table 5. Results of the analysis of variance of wood samples in liquefaction.

Source of Variance	F. D.	S. S.	S. M.	F. V.
Acid concentration (%)	2	2960.2791	1480.1395	424.48*
Error	6	20.9216	3.4869	
Total	8	2981.2007		
PEG 400-glycerin/cotton burr (Ratio)	2	10938.8315	5469.4157	1074.73*
Error	6	30.5346	5.0891	
Total	8	10969.3661		
Pore size (Mesh)	3	4.1542	1.3847	4.35**
Error	8	2.5472	0.3184	
Total	11	6.7014		
PEG 400/glycerin (Ratio)	3	215.8010	71.9337	116.00*
Error	8	4.9610	0.6201	
Total	11	220.7620		

Notes: F. D.- degrees of freedom; S. S.- sum of squares; S. M.- mean of squares; F. V.- F value; * - 1%; and ** 5% significance level

Table 6 shows the percentages of the residue content of cotton burr after liquefaction with the mixture of PEG-400/glycerin at different acid concentrations, PEG 400-glycerin/cotton burr ratios, pore sizes, and PEG 400/glycerin ratios. The percentages of residues of the cotton burr decreased from 55.44% to 12.17% when the acid concentration increased from 5% to 9%. In addition, the percentages of unliquefied cotton burr decreased from 12.10% to 10.61% when the pore size grew from 40 mesh to 100 mesh. However, the percentages of residues of the cotton burr surged from 10.78% to 95.31% when acid concentration increased from a 3-1/10 ratio to a 3-3/30 ratio. The percentages of the unliquefied cotton burr increased from 9.94% to 20.78% when the pore size were enlarged from a 1/1 ratio to a 10/1 ratio.

The amount of unliquefied cotton burrs remarkably decreased with increasing of acid concentration, PEG 400-Glycerin/Cotton Burr ratio, pore size and PEG 400/Glycerin ratio.

Table 6. Groups and mean values in liquefaction of cotton burrs

Acid Concentration		PEG 400-Glycerin/Cotton Burr Ratio		Pore Size		PEG 400/ Glycerin	
(%)	Mean	(Ratio)	Mean	(Mesh)	Mean	(Ratio)	Mean
5	55.44 a	3-1/10	10.78 c	40	12.10 a	1/1	9.94 c
7	25.12 b	3-2/20	63.56 b	60	11.15 ab	4/1	17.17 b
9	12.17 c	3-3/30	95.31 a	80	10.71 b	7/1	19.80 b
				100	10.61 b	10/1	20.78 a
Means	30.91		56.55		11.14		16.92
S_x	1.64		1.82		0.54		0.74
CV	6.04		3.99		5.06		4.65

Notes: a-c: The difference between the average value of the same group with different letters in the same row

Differences in amount of the residue content of cotton burr after liquefaction can be categorized by distances in structures and the types of hemicellulose and lignin of softwood and hardwood. While hardwood lignin is composed of syringyl and guaiacyl units, softwood lignin consists of only syringyl units, which makes hardwood more vulnerable to destructive extraction during liquefaction (Sarkanen & Ludwig, 1971; Alma et al., 2003).

According to Kurimoto et al. (1999), the best reaction result was found to become PEG/Glycerin: 7/3 ratio, 90 min to 120 min in Radiata pine). Alma et al. (2002) found that the amount of material that did not react was the lowest with 120 min in cotton stalks. They determined that as the time increased,

the amount of substances that did not react decreased. Alma et al. (2003) found that the amount of material that did not react was detected the lowest with 120 min in chestnut. Alma et al. (1995) stated that as time increased, the amount of substances that did not react decreased. Yan et al. (2008) indicated that 180 min was the best amount of unreacted material in corn stalks and 170 °C and 190 °C for 2 h were the best temperatures.

The liquefaction yield of the cotton burr remarkably increased with increasing of the liquefaction time, which was attributed to the decomposition of amorphous zones of lignin, hemicellulose and cellulose as they are vulnerable to the liquefaction process (Zhang et al., 2012; Huang et al., 2017).

The data obtained in this study show similarities with published findings (Kurimoto et al., 1999; Alma et al., 2003; Yan et al., 2008).

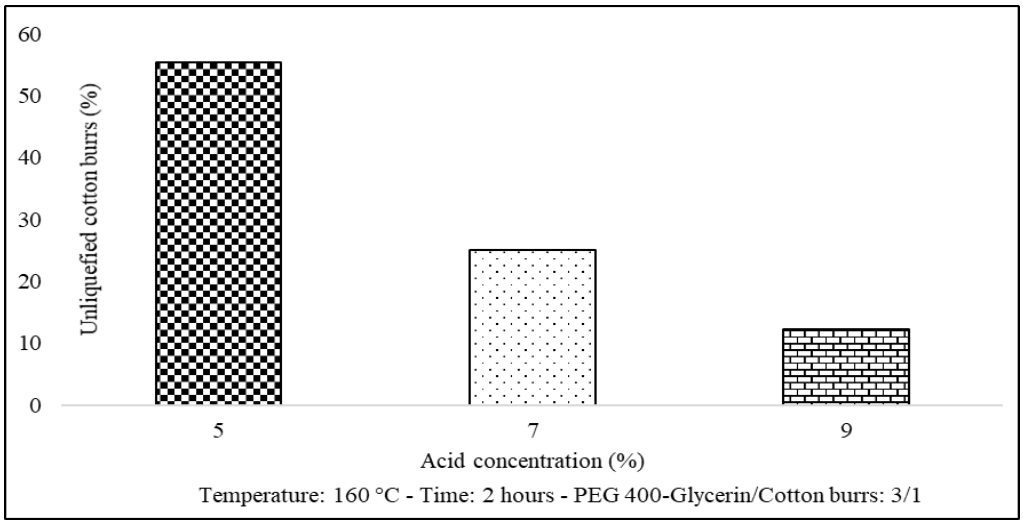

Figure 3. Relationship between the acid (H_2SO_4) concentration (%) and the percentage of unliquefied cotton burrs

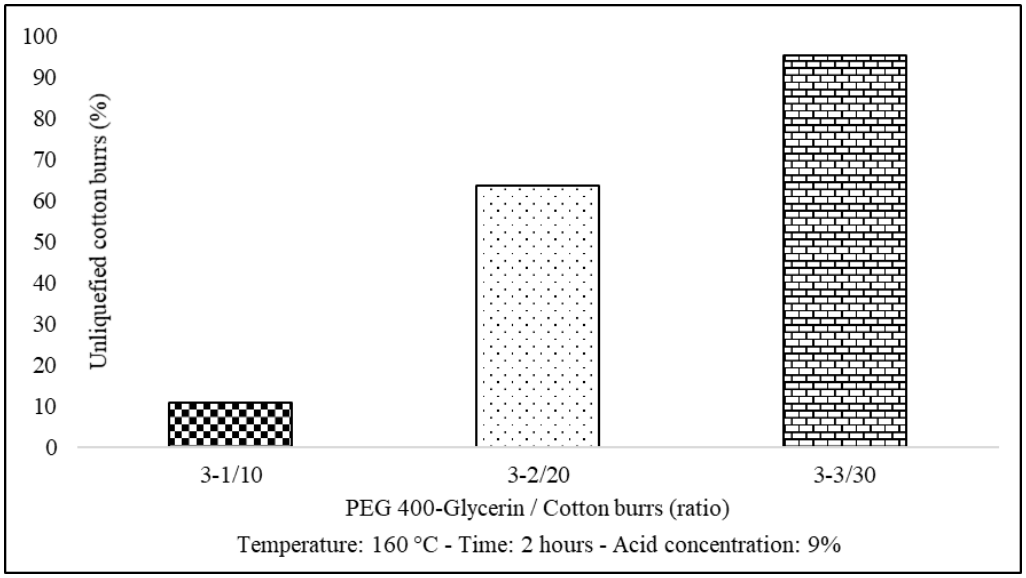

Figure 4. Relationship between the PEG 400-glycerin/cotton burrs (ratio) and the percentage of unliquefied cotton burrs

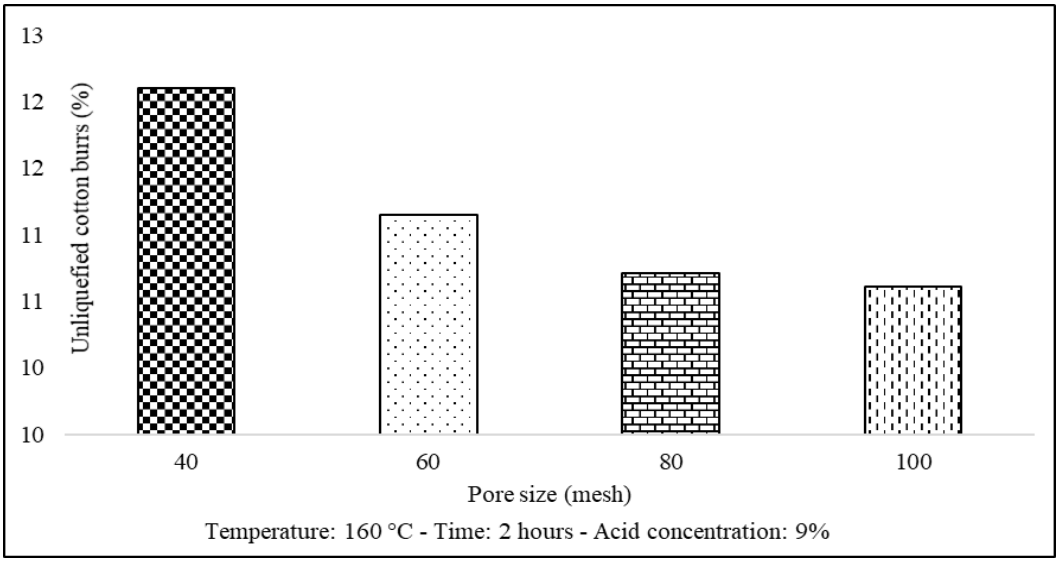

Figure 5. Relationship between the pore size (mesh) and the percentage of unliquefied cotton burrs

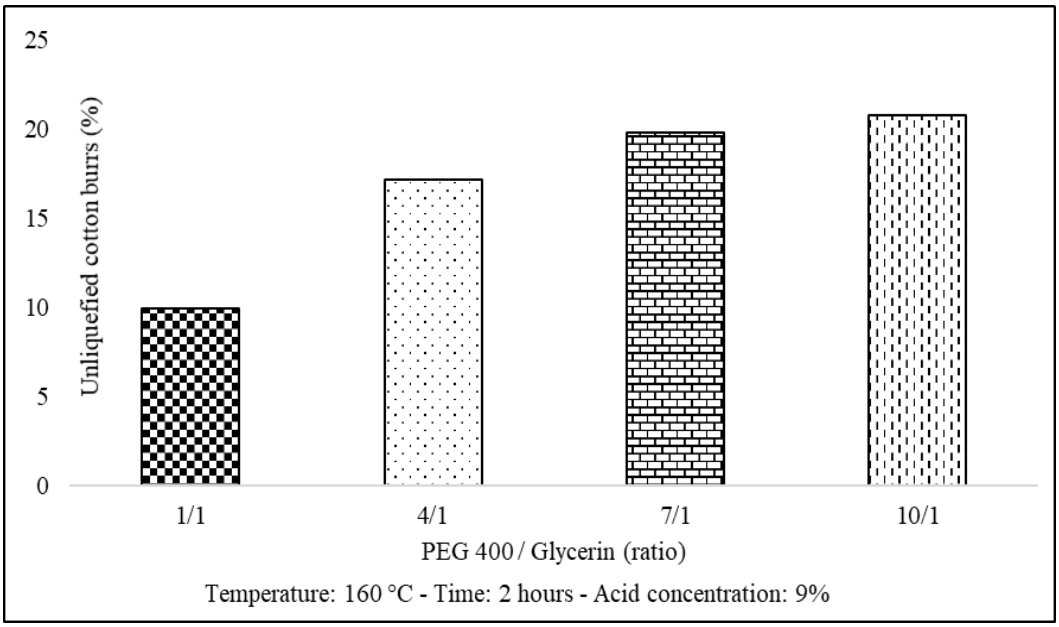

Figure 6. Relationship between the PEG 400/glycerin (ratio) and the percentage of unliquefied cotton burrs

Figure 3-6 illustrate the cotton burr samples liquefied with different acid concentrations, PEG 400-glycerin/cotton burr ratios, pore sizes, and PEG 400/glycerin ratios. The maximum amount of liquefaction in cotton burr was 9% within the group of acid concentrations, a 3-1/10 ratio within the group of polyethylene glycol-glycerin/cotton burr ratios, 100 mesh within the group of pore sizes, a 1/1 ratio within the group of polyethylene glycol/glycerin ratios. Thus, the best of foams obtained with the liquefaction of cotton burrs was with the highest percentage of liquefaction.

The interaction effects among the acid concentrations, the PEG 400-glycerin/cotton burr ratios, pore sizes, and PEG 400/glycerin ratios are reported in Table 7.

Table 7. Multiple correlation analysis of the acid concentrations, peg 400-glycerin/cotton Burrs ratios, pore sizes, and the peg 400/glycerin ratios

	A	B	C	D
A	-	-	-	-
B	-0.79*	-	-	-
C	0.53	0.06	-	-
D	-0.77*	0.98*	0.09	-

Notes: A: Acid concentration (%), B: PEG 400-glycerin/cotton burrs (ratio), C: pore size (mesh), and D: PEG 400/glycerin (ratio)

The correlations between the polyethylene glycol-glycerin/cotton burr ratios and the polyethylene glycol/glycerin ratios were both positive and significant (r = 0.98*); there was a positive correlation between the acid concentration and pore size (r = 0.53). However, between the acid concentration and the polyethylene glycol-glycerin/cotton burr ratio or the polyethylene glycol/glycerin ratio, the correlation was determined as negative (r = -0.79* and r = -0.77*, respectively).

Conclusion

In this study, cotton burr wastes were successfully liquefied with a constant amount of PEG 400-glycerin and at various concentrations of H_2SO_4, which was used as a catalyst. The best result in the liquefaction of cotton burrs for foams was found in the polyethylene glycol/ glycerin (1:1 ratio) with 9% H_2SO_4 for 2 h at 160 °C. The amount of unliquefied cotton burrs significantly decreased with the increasing of PEG 400-Glycerin/Cotton Burr ratio (3-1/10, 3-1/20, 3-1/30), PEG 400/ Glycerin ratio (1/1, 4/1, 7/1, 10/1), pore size (40, 60, 80, 100 mesh) and acid concentration (5%, 7%, 9%). A multiple correlation analysis indicated a highly positive correlation between the polyethylene glycol-glycerin/cotton burr ratio and the polyethylene glycol/glycerin ratio, whereas a highly negative correlation was observed between the acid concentration and the polyethylene glycol-glycerin/cotton burr ratio or the polyethylene glycol/glycerin ratio. Samples within the group of polyethylene glycol/glycerin (1:1 ratio) were significant in the liquefaction process. Thus, the best foams obtained with the cotton burr liquefaction is the highest percentage of liquefaction.

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