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The Effect of Maximum Reaction Temperature of Polyurethane Foam on the Effective Thermal Conductivity

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Abstract

The thermal conductivity of polyurethane foams may be sensitive to many pronounced parameters related to their components and conditions of formation. This study aims to investigate the effect of maximum reaction temperature during forming the foam on the resultant thermal conductivity value. The study used samples from different cases and conditions to monitor the reaction. The cases include standard, with n-pentane or water as blowing agents and ethylene glycol as activation agents. Two conditions have been selected: with and without post-heating. The results have shown that thermal conductivity increases with the increase in the maximum reaction temperature for certain cases (1 g and 2 g). However, for 4 g, there is a decrease in the k-value. In general, the lowest k-value has been noticed for the case of using 1 g of n-pentane (0.033 W/m.K) compared to the reference case that does not use further addition of agents (0.043 W/m.K). The variation of the k-values due to the increasing mass of the agents shows an increment behavior.

Keywords: Blowing agent; Ethylene glycol; Polyurethane; Reaction temperature; Thermal conductivity.

1. Introduction

Polyurethane (PU) contains various organic cyanate esters, polyol groups, and other combinations. PU material has the advantage of thermal insulation and lightness. It is an affordable material, one of the important features distinguishing PU from different polymer materials [1]-[3]. Among them, PU porous materials are the most widely used materials, accounting for more applications of PU. Due to the advancement of science and technology, the use of PU foam materials in thermal insulation has increased in engineering, aerospace, and other industries. Their high performance makes them useful as dielectric material in electrical instruments. Polyurethane's light weight and high performance make it an important part of many industrial applications; it increases performance by reducing fuel consumption, improving safety, and enhancing the comfort zone. Soft foam is widely used in the automotive and furniture industries [4]-[6]. The performance of polyurethane foam depends mainly on cell type, size, and arrangement. Insulation foam manufacturers create closed-cell foam to satisfy the best heat insulation performance. They also develop better sound- and water-proofing features [7], [8].

After the development of synthetic polymers, many types of PU have been made, such as rigid, flexible, and semi-flexible. Rigid polyurethane foam (PU) has been developed recently to include many components and agents besides their basics (polyol and cyanate). The PU foams are more specialized as heat-resistant foams, of which polyurethane-modified isocyanurate foams have reached commercial importance. In general, several recent studies have investigated aspects of the thermal conductivity of polyurethane foam (PUF) related to many parameters, such as type of polymer, rate of contents, type of additives, water content, and processing conditions. For this reason, the value of thermal conductivity may differ according to many physical conditions, like the structure of the foam, bulk density, temperature, pressure, moisture content, and material aging [9], [10]. Several review studies [11]-[13] have presented a general overview of the features of polyurethane foam, especially the thermal conductivity value, with many ideas to improve the capability of PU for thermal insulation under many conditions. The studies offered a lot of data on the thermo-physical properties of PU.

By reviewing the available studies within this field, it can be noticed that the majority of the works focused on the effect of blowing agents on the k-values [14]-[18] or in other thermal properties [19], [20]. Some researchers investigated the effect of many contents in enhancing the foaming of PU, such as iso-cyanate [21]-[24], ethylene glycol [25], [26], nanoparticles [27], [28], and soft segment [29]. Regarding the temperature effects, some studies investigated the variation of k-values in different operating temperatures [30]-[32], and only a few studies [33], [34] studied the effect of reaction temperature on the features of the resultant polyurethane foam. However, the latest studies concentrated on the change in thermo-physical properties in general.

The aim of the current study is to investigate the direct effect of reaction temperature, as an important parameter, on the obtained thermal conductivity value of RPF. Even the previous studies limited the work on the relation between reaction temperature and thermal conductivity value. Still, it is difficult to assign the range of temperature at which the k-value of the PU can be based. The current work contributes to the specific investigation regarding this critical parameter. The novelty of this work is the consideration of maximum reaction temperature as a reference value since the majority of the reaction can be set on and linked to the resultant thermal conductivity value.

2. Materials and Methods

Lab work has been done to investigate the effect of the reaction temperature during the foaming of PU on the effective thermal conductivity value by comparing the obtained data for several manufactured samples under various conditions. The reaction temperature gives an idea about the features of the production as well as the time needed for foaming. Thermal conductivity is the main parameter considered in the current study. The main devices that were used in the study were an electrical oven, a digital thermometer, a thermal conductivity meter, an electrical mixer, and a balance.

The works has been conducted experimentally in Chemical Lab, Materials Engineering Department-Mustansiriyah University. The work included preparing basic components of PU and involving some blowing (or activation) agents within the foaming process to manufacture the desired polyurethane foam for the current investigation. Rigid polyurethane can be formed by mixing multiple organic cyanates and polyol compounds with other additives in the presence of blowing or activation agents. For this study, polyol and polymeric methylene diphenyl diisocyanate (PMDI) were provided by Henkel Polybit Industries Ltd., UAE. The polyurethane can be formed directly by combining the two components, the polyol (A-side) and methylene diphenyl diisocyanate (B-side). The physical and chemical properties of the polyol and isocyanate are listed in Table 1.

Table 1. Specifications of polyol and PMDI*

Property	Polyol	PMDI
Specific gravity at 25°C	1.16	1.22-1.25
Viscosity at 25°C	Approx.450 cps.	150 - 200 cps
NCO% Wt	-	30-31
Acid content (HCL)	-	< 0.05%
Hydrolysable chlorine	-	< 0.2%

* Supplied by the datasheet.

The study proposed to seek the effect of adding extra blowing agents on the behavior of the reaction of PUF. For this purpose, some blowing agents, such as n-pentane and water, have been used. LABORAT GMBH BERLIN provided the n-pentane. The characteristics of n-pentane are presented in Table 2. The blowing agents have been mixed with polyurethane components in suitable masses (1-4 g).

Table 2. Properties of n-pentane*

Properties	Description
Appearance	Colorless liquid
Molecular weight	72.15 g/mol
Melting point	−130.5 to −129.1 °C

* Supplied by the datasheet.

Pure ethylene glycol (EG) has been used as an activation agent (alcoholysis agent). It has been mixed with polyol as a control factor in the polyurethane synthesis with different masses (1-4 g). The primary characteristics of ethylene glycol are listed in Table 3.

Table 3. Properties of ethylene glycol*

Properties	Description
Appearance	Clear and colorless liquid
Molecular weight	62.07 g/mol
Melting point	-12.9 °C
Density	1.11 g/cm³

* Supplied by the datasheet.

In the current study, a rectangular wooden mold with 13.4 x 13.4 x 21.1 cm^3 was formed to form the foam. An aluminum foil has been lining from the internal surface to make the unfolding easier. Four processing steps have been applied for the molding of polyurethane foam, as follows:

Step 1. Weight 50 g of the A-component material in a plastic cup. Add 50 g of the B-component to a syringe to avoid material loss. The blowing agent (or activation agent) can also be added to a plastic cup and mixed with it.

Step 2. The combination (A-component, B-component, blowing agent, catalyst, and surfactant) was blended using an electric mixer at 1500 rpm for 10 seconds.

Step 3. The mixture was then quickly poured into a rectangular wooden mold to form the PU foam and measured the reaction temperature using a thermometer with a sensor inserted into the mold, as shown in Fig 1. Temperature and time were recorded for several minutes until the foaming was complete.

Step 4. Remove the PUF block from the mold (Fig 2). Cut the block into several square samples (5 cm x 5 cm x 1 cm) to be ready for measurements, as shown in Fig 3.

Figure 1. Measurement of reaction temperature

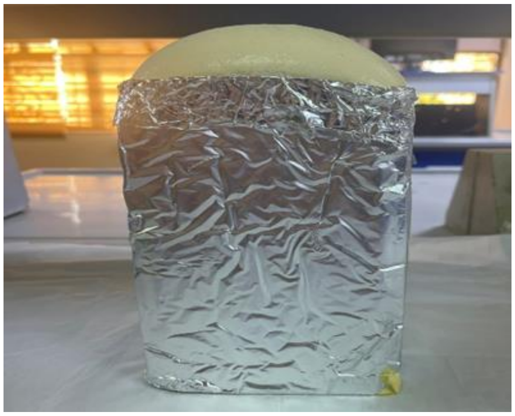


Figure 2. Block of PUF

Figure 3. Sample of PUF ready for measurements

The foaming of PU is completed in 3-5 min at room temperature [35], [36]. Therefore, the samples have been tested directly after foaming. However, the setting time for the foamed material to be completed rigid may take several days [17]. Initially, it is important to determine suitable mixing conditions (time and speed). Studies recommend mixing time between 5-20 s and mixing speed between 500-2000 rpm [23], [33]. Therefore, these factors have been considered before any other factors. After fixing the suitable mixing conditions, 10 s for mixing time and 1500 rpm for mixing speed, the study focused on the role of post-heating. The heating was done by putting the foam in the oven at 60°C for 30 minutes. The physical blowing agents or controlling agents were used in a range of content by mass, as 1, 2, and 4 g, and mixed with the A & B components in a ratio of 50:50. Note that it was difficult to form the PUF at 4 g addition of water because the reaction has not completed. The reaction rate of isocyanate and water is much faster than the polymerization reaction. This generates a high amount of gas before enough polymer is formed to hold the gas in the cell. Thus, the bubbles escape during the reaction. A suitable amount of water is selected for the polyurethane foam reaction [37].

Different samples have been prepared for various cases and conditions. The cases include the type of materials used, such as neat foam (components A & B without any addition), with n-pentane, with water, with EG, and with both n-pentane and EG. The conditions include different contents of the agents and different heat circumstances, such as with or without post-heating of the specimen. The cases and conditions are all listed in detail in Table 4.

Table 4. Details of cases and conditions under study

It.	Case	Mass of agent (g)	Condition of heat
A1	Without any addition	-	-
A2	Without any addition	-	Post
B1	With n-pentane	1	-
B2	With n-pentane	1	Post
B3	With n-pentane	2	-
B4	With n-pentane	2	Post
B5	With n-pentane	4	-
B6	With n-pentane	4	Post
C1	With water	1	-
C2	With water	1	Post
C3	With water	2	-
C4	With water	2	Post
D1	With EG	1	-
D2	With EG	1	Post
D3	With EG	2	-
D4	With EG	2	Post
D5	With EG	4	-
D6	With EG	4	Post
E1	With n-pentane & EG	1	-
E2	With n-pentane & EG	1	Post
E3	With n-pentane & EG	2	-
E4	With n-pentane & EG	2	Post
E5	With n-pentane & EG	4	-
E6	With n-pentane & EG	4	Post

Note that the cases represent selected ones from a wide range of materials and conditions that have already been tested. They satisfied the formation of low-density rigid PU foams without shrinkage or crumbling.

As presented in Fig 4, thermal conductivity values were measured using a MED-103 thermal conductivity meter. The experiments were performed according to the ASTM C177 standard (Direct Absolute Technique), in which the sample was square with an area of 25 cm^2 and 1 cm thick.

Figure 4. Device used to measure thermal conductivity

The device includes an enclosure and an electrical box. The enclosure contains an electric heater where the samples should be placed with appropriate partitions. The circle should be opened until thermal balance is achieved. The reader records the temperatures using sensors connected to the data logger. Note that these readings should be substituted in the Fourier Equation as the general equation that regulates heat transfer in materials [38].

3. Results and Discussion

The current study's results are limited to rigid polyurethane foam (RPF) composed directly by mixing the basic components (polyol and PMDI) with the addition of some agents under certain thermal conditions. The results present the variation of reaction temperature, reaction time, and thermal conductivity for the selective cases.

3.1. General results

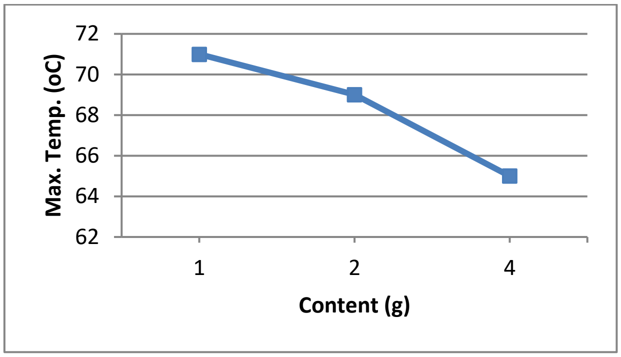
Initially, the results for the standard case (without any addition) are shown in Table 5. These values are considered as reference points for comparisons with other cases. The results show that the maximum reaction temperature can reach up to 129 C with an average time duration of about 7.5 min. The k-value is stated at 0.045 W/m.K, which is within the highest limit for RPF, where the common k-values are usually less than that by 10-20% [30], [32], [39], [40]. However, the error is due to the extremely low k-value of PU, which is difficult to satisfy with conventional instruments. This is attributed to the high frequency of the voltage and current in the thermometers and the electrical board readers, which affect the heat rate value. Furthermore, the results show that the k-value is even lower in the case of post-heating. The reason behind this is the reduction in the water content, which minimizes the high thermal conduction caused by water molecules and increases the voids [41]. Moreover, the densities of the polymer decreased while porosities increased due to weight loss associated with the water escape as a result of heat treatment [42]. The current samples are considered low-density polyurethane foams with 10-30 kg/m^3. Also, the obtained k-values may save energy by 20-40 W/m^2 (30-60%), depending on the one-dimensional Fourier Law, to satisfy comfortable indoor conditions in summer.

Table 5. Results for the standard case

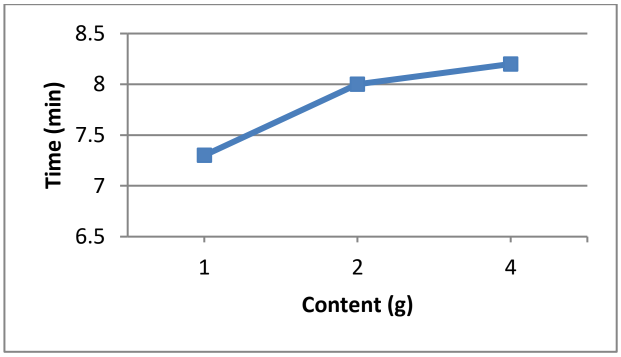
It.	Case	T-max (ºC)	Time (min)	k-value (W/m.K)
1	Standard without post-heating	129	7.5	0.045
2	Standard with post-heating	129	7.5	0.043

The results for other cases using n-pentane, water, EG, and both n-pentane & EG are shown in Figs 5 to 8. The results have shown lower reaction temperatures when using these additional agents compared to the reference case (without addition). However, a higher T_{max} was noticed for the case of using EG (up to 116 ºC) compared to other cases, then water (up to 90 ºC), then n-pentane with EG (up to 82 ºC), and lastly, the case of n-pentane (up to 71 ºC). With respect to the variation of maximum reaction temperature (T_{max}) based on the mass content of the n-pentane agent, there is a decrease in the T_{max} by increasing the mass of the blowing agent from 1g to 4 g by 8.4 %. When mixing n-pentane with EG, the total decrease in the T_{max} was 10.9 %. In the case of using water, the reduction in the T_{max} was 11.1 % when the mass of the agent increased from 1g to 2 g. However, for the case of using EG only, there is an increase in the T_{max} by the increase of the mass from 1g to 4 g by 22.1 %.

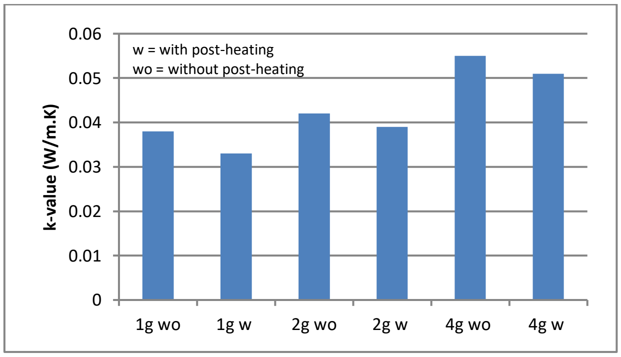
The results have shown that it takes less time to reach the maximum reaction temperatures when using the additional agents compared to the reference case (without addition). However, the longest time was noticed for the case of using n-pentane (7.3 min) compared to other cases at 1 g loading, then water (7.2 min), then n-pentane with EG (6.4 min), and lastly, the case of EG (5.1 min). This sequence is opposite to that observed for the case of T_{max}. In general, the variation of the time required to reach the maximum reaction temperature due to the increasing mass of the agent shows an increment behavior. There is a total increase in the time by 12.3 %, 25.5 %, and 26.5 % for the case of using a higher content of n-pentane, EG, and n-pentane with EG, respectively. But for the case of using water, there is a decrease in the time by 13.3 % due to the increasing mass of water from 1 g to 2 g.



A. Maximum reaction temperature

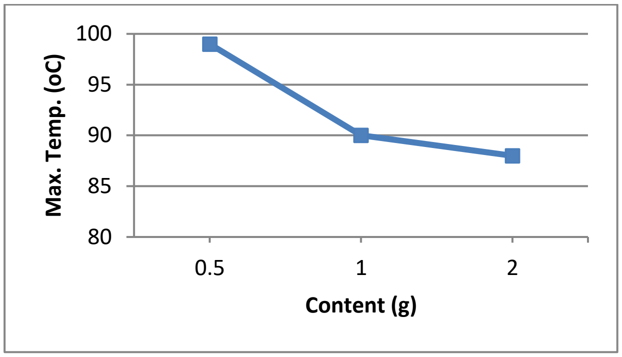


B. Duration to reach maximum temperature

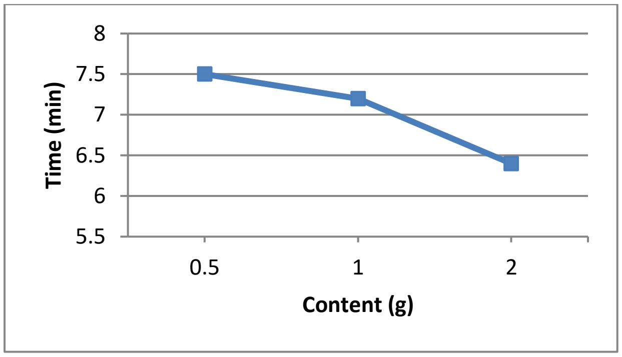


C. Thermal conductivity

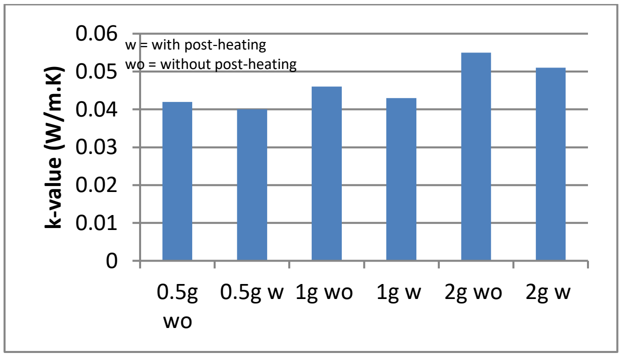
Figure 5. Results for the case of using n-pentane



A. Maximum reaction temperature



B. Duration to reach maximum temperature



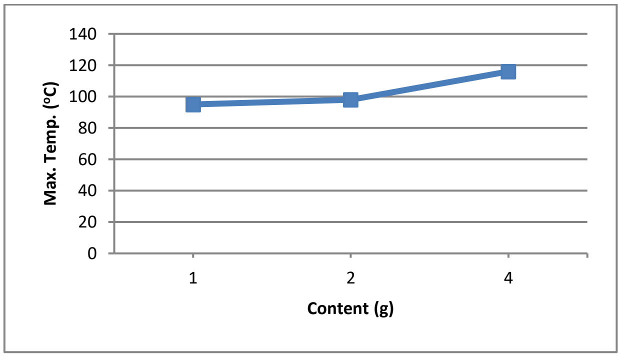
C. Thermal conductivity

Figure 6. Results for the case of using water

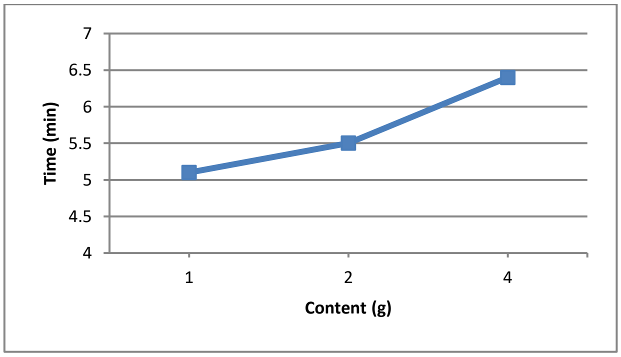
Regarding thermal conductivity, the results showed different values when using the additional agents compared to the reference case (without addition). However, the lowest k-values were noticed when using n-pentane (0.033 W/m.K) compared to other cases. In general, the variation of the k-values due to the increasing mass of the agent shows an increment behavior. There is a total increase of 45 %, 31 %, and 10 % for the case of using a higher mass of n-pentane, water, and n-pentane with EG, respectively. But for the case of using EG, there is a decrease in the k-value by 16 % due to the increase of the mass of water from 1 g to 4 g, but it was still higher than the reference k-value (0.043 W/m.K).

3.2. Effect of the reaction on k-value

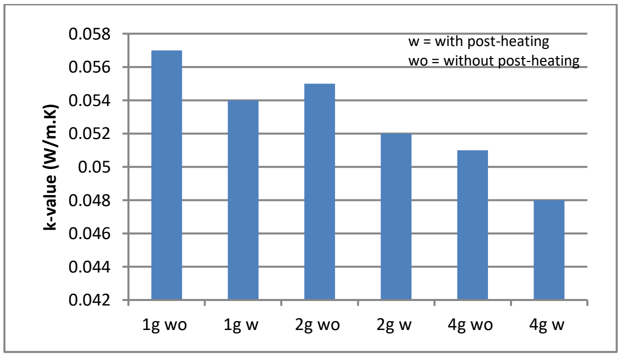
The combined effects of the maximum reaction temperature and the time required to approach the maximum reaction on the corresponding k-values can be shown in Fig 9 and 10. It can be noticed from the overall behavior of the results that thermal conductivity is increasing with the increase of the maximum reaction temperature for some instances (1 g and 2 g). However, for 4 g, there is a decrease in the k-value due to the increase in reaction temperature. Also, the thermal conductivity decreases with the increased time required to reach maximum reaction temperature for certain cases (1 g and 2 g). However, for 4 g, there is an increase in the k-value with time.



A. Maximum reaction temperature

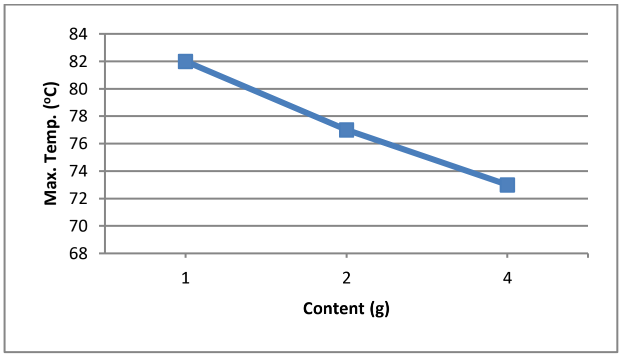


B. Duration to reach maximum temperature

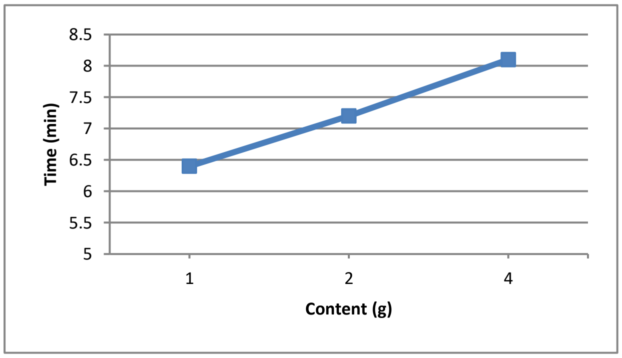


C. Thermal conductivity

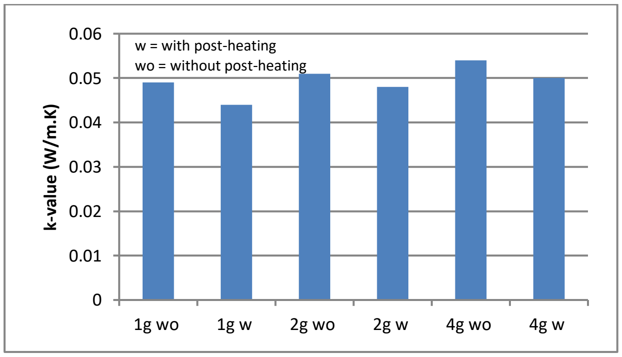
Figure 7. Results for the case of using EG



A. Maximum reaction temperature



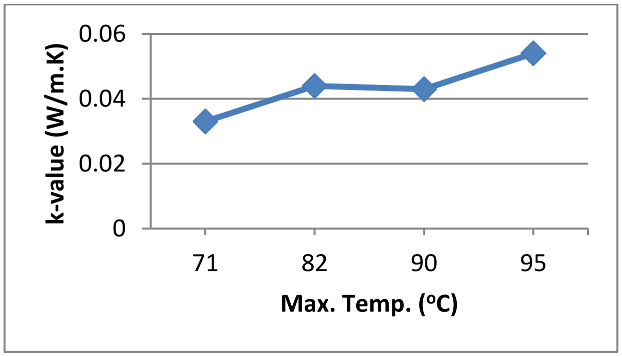
B. Duration to reach maximum temperature



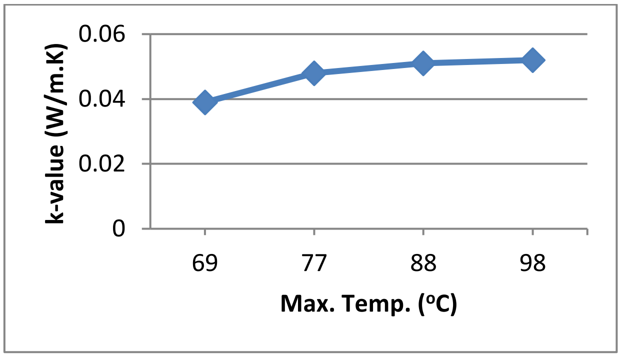
C. Thermal conductivity

Figure 8. Results for the case of using n-pentane & EG

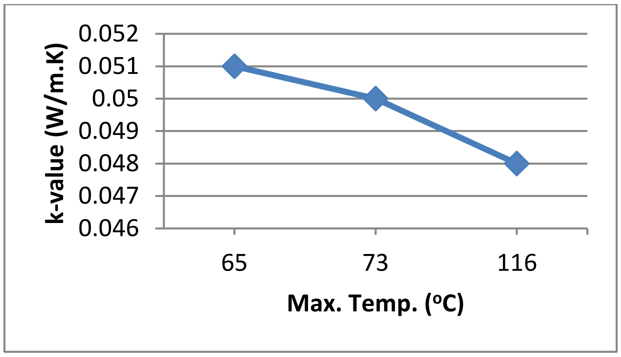
When adding a higher amount of blowing agent, substantial heat was generated due to a more vigorous reaction between the blowing agent and PMDI than with the polyol [15]. This explains why the reaction temperature required for foaming was lower in the case of high loading of agents. This may also increase average cell size and interconnected cells compared to the neat foam. Increasing cell size means increasing convection and radiation heat transfer [32]. Thus, the extra mass of the agent shows an increase in the foam's k-value.



A. Content of 1 g

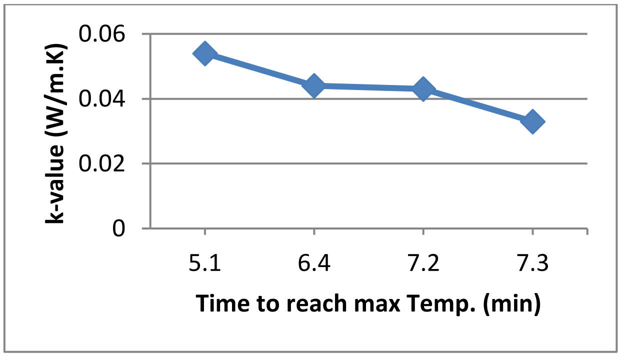


B. Content of 2 g

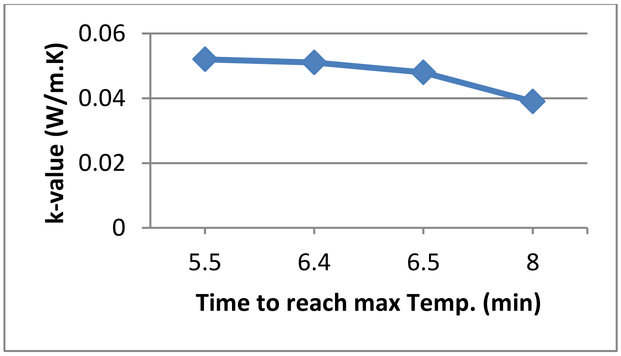


C. Content of 4 g

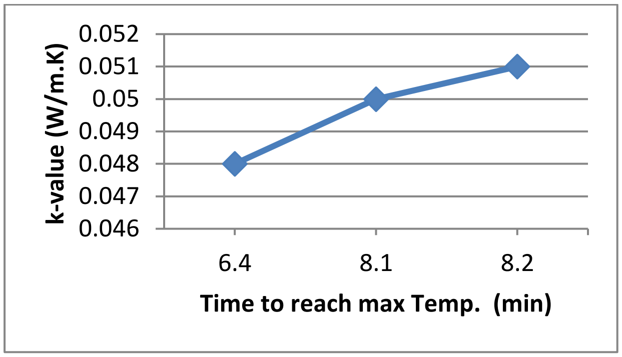
Figure 9. Effect of maximum reaction temperature on the K-value



A. Content of 1 g



B. Content of 2 g



C. Content of 4 g

Figure 10. Effect of the time to reach maximum reaction temperature on the k-value

3.3. Comparison

For validation purposes, an analyzing comparison has been made to see the convergence of the current results with those mentioned by some reliable studies, taking into account the variations in their conditions, as shown in Table 6. In general, the results of the studies are compatible with the current results. Note that some studies show slight differences due to the variations in the features of PUF and operational conditions.

The main findings that can be recognized to have the same behavior for both current and previous works are the following:

- The increase in the blowing agent, to some extent, leads to obtaining PUFs with lower thermal conductivity values. This still works for small cell sizes.
- Usually, the lower the foam's density, the lower its thermal conductivity value. However, this is not always true due to the effect of cell size. For higher cell sizes, convection and radiation heat transfer increase; thus, there is a possibility of an increase in the k-value.
- However, the decrease in density certainly reduces the solid thermal conductivity of the effect k-value due to the rise in porosity.

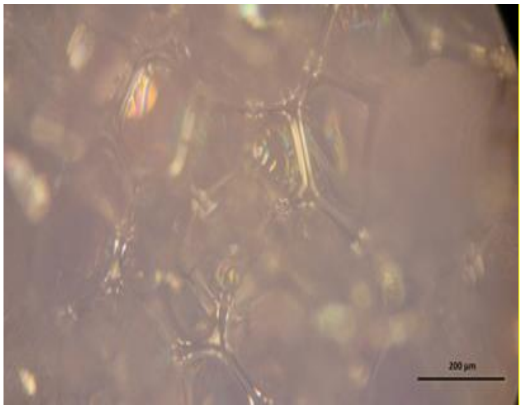
3.4. Morphological considerations

To check the variation in morphological features due to the investigation under several cases and conditions, microscopic images have been captured by the microscope (MT7100) for certain polyurethane foam samples. The microscopic images of the PU foam for selected specimens under certain cases are shown in Fig 11. It can be recognized that the cells are mostly spherical or elliptic, with plane sides that take polygonal shapes, such as hexagonal, heptagonal, or octagonal. In some cases, it has an oval shape. Depending on the case, the number of cells in the image may differ from 20-30 units. Also, cell size can be between 50-500 µm based on the conditions. The cells of PU foams show variation in wall thickness from 20-35 µm.

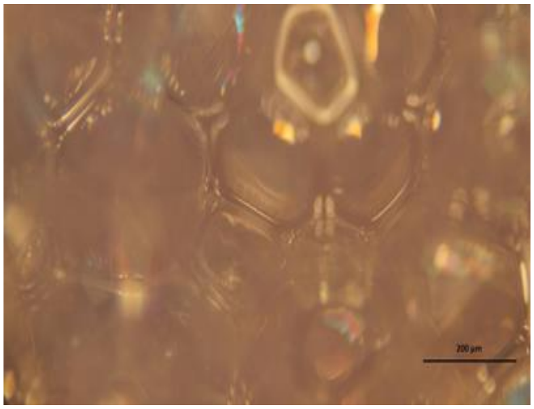
Table 6. Comparison and comments on the findings of some corresponding and reliable studies

Ref.	Findings	Comments
[15]	The study indicates that PUFs containing more blowing agents have lower thermal conductivity.	This is due to the decrease in cell size compared to the neat foam. However, this behavior can be seen in the current work b adding 1 g of the foaming agent.
[18]	Lowering the density of the foam decreases its thermal conductivity.	The decrease in k-value due to the decreasing density is not always true due to cell size effects. For higher cell sizes, convection and radiation heat increase, so there is a possibility of an increase in k-value.
[19]	The type and amount of the blowing agent impact foam density by expanding the cells. More blowing agent tends to create lower-density foam, which impacts foam properties such as thermal conductivity.	For the current study, the type and mass of the blowing agent affect the density by the same behavior as the mentioned findings until a certain amount of addition is made.
[22]	In the case of PUR foams, smaller cell sizes lead to lower thermal conductivity.	Smaller cell size is essential to satisfy lower k-values.
[31]	Thermal conduction usually increases as the density increases	The increase in k-value is attributed to the increase in the solid component.
[32]	As the foam's density decreases, the thermal conductivity of the gaseous phase and heat transfer by radiation increase, while the thermal conductivity of the solid matrix decreases.	This is precisely what happened in the current work. The increase in k-value is due to the increase in convection and radiation modes of heat. The decrease in the solid part reduces the solid thermal conduction due to increased gas in place of the solid within the matrix.

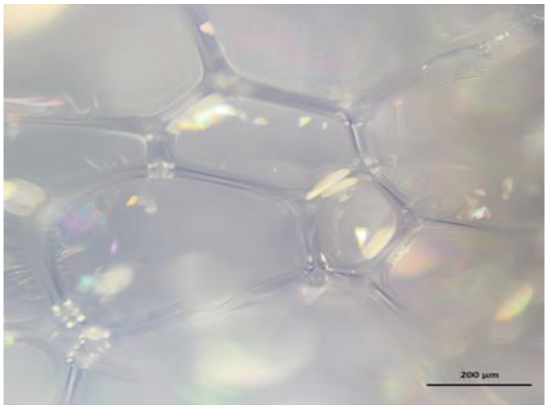
In general, the cells' quantity and size change due to the procedure that occurred, such as with or without the post-heating, as well as the amount of blowing agent added. In the case of post-heating, the cells remain spherical but with more voids. This explains why post-heating showed less k-value due to high porosity. The cells in foam subjected to the extra amount of the blowing agent have elliptic forms with fewer units and larger sizes. Therefore, it showed a higher k-value. The increase in the effective thermal conductivity in big cell sizes is due to the rise in the modes of heat transfer by convection and radiation [32].



A. Standard PU without post-heating



B. Standard PU with post-heating



C. PU with extra n-pentane (4g)

Figure 11. Microscopic images for selected cases of PU foam with 200 μm scale

4. Conclusions

The current study focused on the direct relationship between the thermal conductivity and the maximum reaction temperature of the PU composed directly or by adding extra blowing agents. The results show the following behaviors: The k-values were within the highest limit for RPF. The k-value is lower in the case of post-heating. In general, the variation of the k-values due to the increasing mass of the agents shows an increment behavior. The lowest k-value was noticed when using 1g of n-pentane (0.033 W/m.K) compared to the reference case (0.043 W/m.K). The thermal conductivity increases with the increase of the maximum reaction temperature for certain cases (1g and 2g). However, for 4g, there is a decrease in the k-value. The thermal conductivity decreases with the increase of the time required to reach maximum reaction temperature for certain cases (1g and 2g). However, for 4g, there is an increase in the k-value with time. The maximum reaction temperature and the duration to reach it were lower when using additional agents compared to the reference case (without addition).

However, many research directions can be suggested for future works, such as using other blowing agents to investigate the role of reaction temperature on thermal conductivity values. Also, the preparation of PU foam under ambient conditions exhibited considerable shrinking. Preheating and using polyols with higher hydroxyl numbers can be solutions.

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Abbreviations

EG	Ethylene Glycol
PMDI	Polymeric Methylene Diphenyl
PU	Polyurethane
PUF	Polyurethane Foam
RPF	Rigid Polyurethane Foam

Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

Author Contribution Statement

Masarra A. Fadhil is a master's student. Tawfeeq W. Moahmmed and Harith H. Al-Moameri are the supervisors of this research. They proposed the research problem and supervised the findings of this work.

All authors discussed the results and contributed to the final manuscript.

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