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Investigation of the Polyol Types and Isocyanate Concentrations on the Rheological, Morphological and Mechanical Properties of Polyurethane Foams

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Investigation of the Polyol Types and Isocyanate Concentrations on the Rheological, Morphological and Mechanical Properties of Polyurethane Foams

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Abstract. This research studies the preparation and characterization of polyurethane foams, which are produced from the reaction of isocyanate Methylene Diphenyl Diisocyanate “MDI” and polyol with water being used as a chemical blowing agent. The effect of isocyanate concentration on the cross-linking formation of polyurethane samples and its influence on the final properties of the foams were investigated. Each type of polyols (Local commercial market polyester, Quickmast120 and Quickmast110 polyether) were mixed with the isocyanate at equivalent ratio of (isocyanate: polyol) (0.5:1, 1:1, 2:1 and 3:1) to prepare polyurethane foam using one shot method. Rheological tests were performed for the polyols and isocyanate to find out their effect on the formability processes. The morphology and structure were performed using FTIR and digital microscopy. The tensile strength, compression strength and the hardness were conducted to show the effect of isocyanate concentrations on the mechanical properties of samples. The result display the Quickmast 120 polyol was unsuitable due to the large cells, unstable rheological properties and high hardness, which leads to a loss of flexibility of the polyurethane foams. The ratio of (1:1) (polyol/isocyanate) was the best in terms of the formability of the foam giving the smaller cells size and the best mechanical characteristics as compared to other concentrations of isocyanate.

Keywords. Polyurethane foam, Cellular structure, Rheological, Mechanical properties, Blowing agent.

1. Introduction

It is durable to go a day without coming crossways some kind of plastic microcellular. Plastic microcellular are established practically all over in our current life and are utilized in an extensive kind of uses such as disposable packaging of fast food, the softening of equipment and insulation materials [1]. Microcellular materials are defined as one that is prepared of inter-connected networks of solid plate that form edge and face of cell [2]. Since 1930s, microcellular plastics have established extensive achievements in engineering and consumer uses due to the low materials cost, higher strengthen, varied kind of characteristic and easiness of processing. Furthermore, these materials are mainly important since they can be produced with usual cell dimensions from few microns to hundreds of microns. For any



specified plastic, utilizing altered blowing agent and processing condition can yield (new material) with changed density, structure, and characteristic [3]. Plastic foaming is a polymer processing technology that involves the use of blowing agents, and sometimes other additives such as nucleating agents, to generate cellular structures in a polymer matrix. It is a complicated phenomenon, which involves thermodynamics, reaction kinetics, melt/gas rheology, gas diffusion and permeation, and others [4]. Polyurethane (PU) is a plastic consists of chains of carbon-based units linked by “urethane” link and are shaped by reactions of isocyanates with polyols. The product is thermosetting foam or elastomer that is straight formed into final equipment [5]. PU foam (frequently referred to as urethanes foam) which is prepared by the reactions of isocyanates with polyols in the presence of water. The reaction of isocyanates with water is strong since isocyanate reacts freely and fast with water, mainly below the influence of catalyst, and to some extent difficult since an unbalanced inter-mediate is first shaped (carbamics acids), with the related growth of CO₂ [6]. Since the 19th century, metals, leathers, and woods were utilized as prosthetic materials. Earlier 1984 lower limb prostheses were synthetic of metals, leathers, or "plastics laminated with foams toe fillers", while, these device did not return functions lost by amputations since it is very stiff [7]. One such is the modification of the Jaipur Foot based on Polyurethane polymers for the benefit of the disabled [8]. In recent years, the PU is one of the most versatile polymers in the modern cellular plastic industry. The majority of PU used in the productions is of flexible foam (48%), followed by rigid foam (28%), elastomer (7.8%) and is also utilized for special applications to range of 16.2% in the forms of coating, fiber, adhesive, caulk, sealant, etc. [9]. This study is dedicated to the preparation and characterization of a cellular polymer using chemical blowing agents that is suitable to use in Prosthetic limb applications (foot). The effect of isocyanate concentration on the cross-linking formation of polyurethane samples and its influence on the final properties of the foams were investigated. Various types of polyols were mixed with the different isocyanate content to prepare polyurethane foam using one shot method. The tensile strength, compression strength and the hardness were conducted to show the effect of isocyanate concentrations on the mechanical properties of samples.

2. Experimental part

2.1 Materials

The materials used in preparation of polyurethane microcellular are mainly two parts; resin (polyol) and hardener (isocyanate). The resin and its hardener were supplied from Don Construction Products (DCP), the Iraqi Local commercial type has also been used. The main specifications of 110 polyols, 120 polyols are shown in Table (1).

Table 1. The main specifications of 110 & 120 polyols.

Specifications	Types	Results
Form	110 & 120	Liquid
Color	110	Colorless
	120	Red Brown
Flash Point (°C)	110	>100
	120	300
Relative Density at 25 °C (g/cm ³)	110	0.957
	120	0.95- 1
Water solubility	110 & 120	Insoluble

The main specifications of the MDI (Methylene diphenyl diisocyanate), hardener that are needed in the preparation of polyurethane microcellular are shown in the Table (2).

Table 2. The main specifications of 110 hardener.

Specifications	Types	Results
Form	110& 120	Liquid
Color	110	Brawn
	1120	Yellowish Green
Relative Density at 25 °C (g/cm ³)	110	1.12-1.16
	120	1.1-1.2
Viscosity (cP) @ 25 °C	110	60 – 120
	120	125- 150
Pot life in absence of water	110	3 – 4 hr @ 25°C
		2 – 3 hr @ 40°C
		35 – 45 min @ 25°C
	120	15 – 20 min @ 40°C
Water solubility		Insoluble
Reaction on time with water	110	5 – 30 sec @ 25 °C
Gel time	120	40 – 50 min @ 25°C
		18 – 25 min @ 40°C
Shore A hardness	120	60 – 90

2.2 Preparation of microcellular samples

Foam was prepared using cup foaming or one-shot foaming process. Equipment used was paper/ plastic cups, stopwatch, medical syringe, stirrer, and weight balance, foaming system was kept at constant temperature (25 °C) at 30 rpm using distilled water (5 drops). Different batches were prepared by changing the amount of raw materials, and types as shown in Table (3) for changing isocyanate concentration.

Table 3. Experimental data for synthesis of polyurethane foam.

Symbol No.	Equivalent ratio of Isocyanates concentrations	Polyol %	Types
1	0.5	1	Quickmast 110
2	1	1	Quickmast 110
3	2	1	Quickmast 110
4	3	1	Quickmast 110
5	0.5	1	Quickmast 120
6	1	1	Quickmast 120
7	2	1	Quickmast 120
8	3	1	Quickmast 120
9	0.5	1	Local commercial
10	1	1	Local commercial
11	2	1	Local commercial
12	3	1	Local commercial

3. Microcellular polymer test

- **FTIR device** Japan's designer "Kyoto Japan" factory by companies of "Shimatzu Corporation" type (IRAFFINITY1) utilized to perform the physical test to check the structures of polyols and isocyanates for each type and its microcellular structure by clarification the main bands in addition to show the bands after reactions between polyols and isocyanate.
- **The morphology** of the foam samples was performed using digital microscopy (model AM415T Dino-lite Edge, Japan) with magnification rate (20x-220x) and image sensor 1.3 Mega Pixel.

- **The dynamic viscosities** of the samples are studied by means of (cone on plate) viscometers DV-III ultra-programmable rheometers (ASTM D2893) with the cone diameter of 4.8cm and cone angles of 3° .
- **The hardness test** is performed according to (ASTM D2240) by Durometer hardness test, type (Shore A) at depressing time of measuring equal to (15 Sec) at room temperature. The surface of specimens must be smooth, also, the depth of indentation measure on scale has graduation from (0 to 100) hardness numbers. The hardness value is very sensitive to the (specimen thickness, specimen diameter and distance from the edge more than 12 mm). Each specimen was tested seven times at different positions of each specimen at the same time and the average value was taken.
- **The tensile test** is performed according to (ASTM D638-03) using tensile machine (universal testing machine), type (Instron) at a cross head speed "strain rate" of 5 mm/min until break of specimen occurs. The foam tensile strength test is similar to testing other polymers, but gripping and measuring extension require a bit more care. The minimum foam thickness during testing is (12.5 mm) and it is common to measure thicker samples. Foams do not typically have high tensile strengths, so low force (1kN) capacity grips are fine.
- **The compression test** is performed according to (ASTM D 1621-16) using the same tensile machine at cross head (strain rate) of (2.5 mm/min) and applied load until break of specimen occurs. Compressive strength of polymeric plastic foam cellular material is determined by applying compression loads to test specimens having a circular cross section. Specimens must be carefully prepared so faces are parallel to each other and perpendicular to the sides.

4. Results and discussion

4.1. Structure and morphology

The structural groups were analyzed using Fourier Transform Infrared (FT-IR) Spectroscopy. Figures (1, 2 and 3) show the main structure of the pure components (local commercial market, Quickmast 110 and Quickmast 120) polyols. The FTIR spectra in Fig. (1) detailed the local commercial market polyols shows all individual peaks. The stretching broad peak at $3483\text{-}3365\text{cm}^{-1}$ indicates the presence of pending (OH) group in the polyester polyols. The main transmission bands at $2925\text{and}2854\text{cm}^{-1}$ are owing to aromatic and aliphatic C-H stretching. The aromatic C-C bond is inveterate by bands at 1599cm^{-1} . The presence of aliphatic C-O linkages in backbone is indicated by $1281\text{ and }1071\text{ cm}^{-1}$. The C-H bending is confirmed by transmission band at 1123 cm^{-1} . Sharp transmission band at 1458 cm^{-1} could be attributed to presence of methyl group in the polymer structure. The C-O group from carbonyl ester is confirmed by transmission band at 1730 cm^{-1} , and benzene ring is represented at 1600 cm^{-1} , 740 cm^{-1} and -NH group around 3331 cm^{-1} , 1530 cm^{-1} . local commercial market is considered to show polyester structure, this is in good agreement with [10 and 11].

The FTIR spectra in Figures (2 and 3) recorded for the Quickmast (110 &120), demonstrate the main peaks as shown below. The characteristic broadening vibration of O-H bond are presented at 3340cm^{-1} , and the appearance of new bands at 1735 cm^{-1} C=O (carbonyl group) also 2972 represented to the C-H. The C-O stretch is found between $(1018.41\text{ and }1375.25)\text{ cm}^{-1}$. Aliphatic ethers give one strong asymmetric stretch around 1163.08 cm^{-1} , and a very weak symmetric stretch around 850 cm^{-1} . The alkyl ethers give two bands around $(1300\text{ and }1055)\text{ cm}^{-1}$, symmetric and asymmetric respectively, 110 Quickmast is considered to show polyether structure, this is in agreement with [12 & 13].

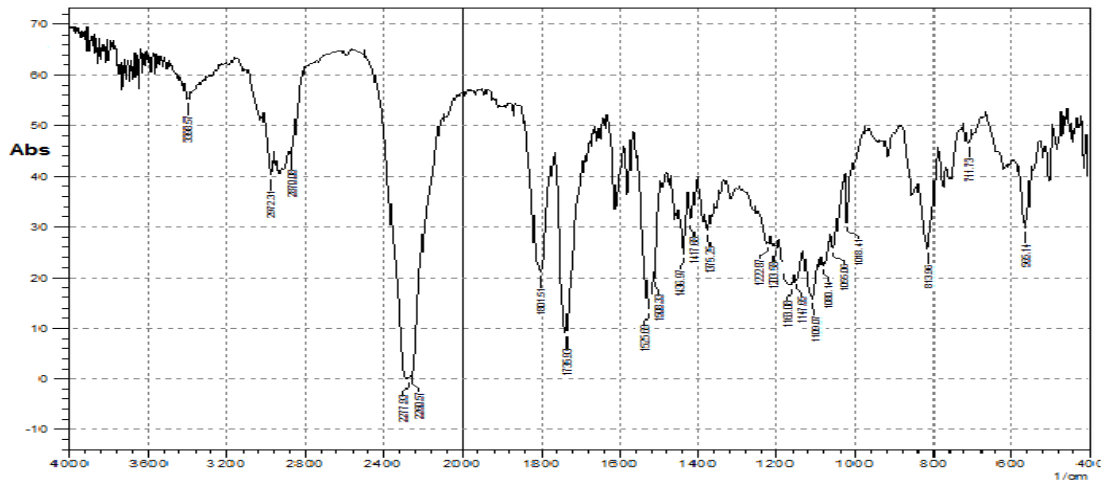


Figure 1. FTIR of (Local commercial market) polyester polyol.

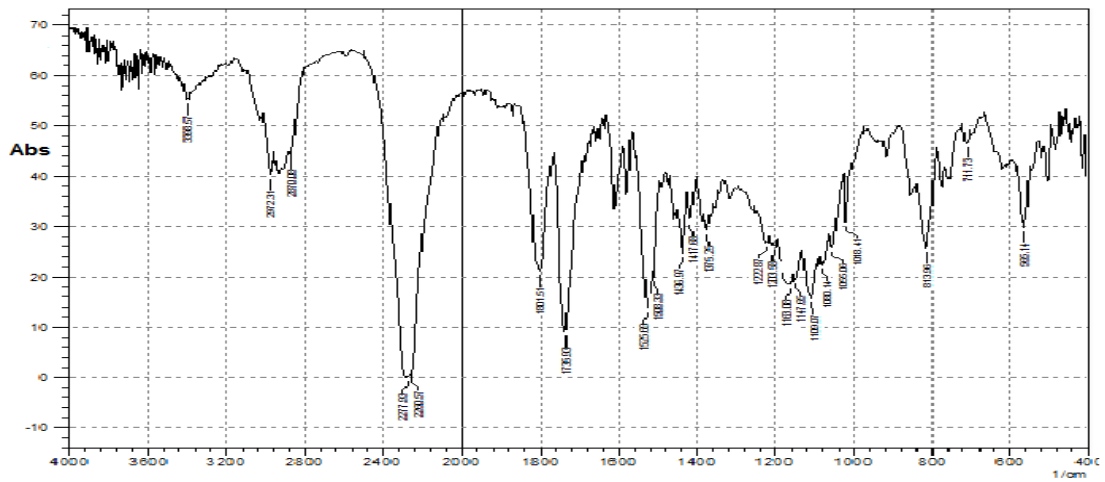


Figure 2. FTIR of (Quickmast 110) polyether polyol.

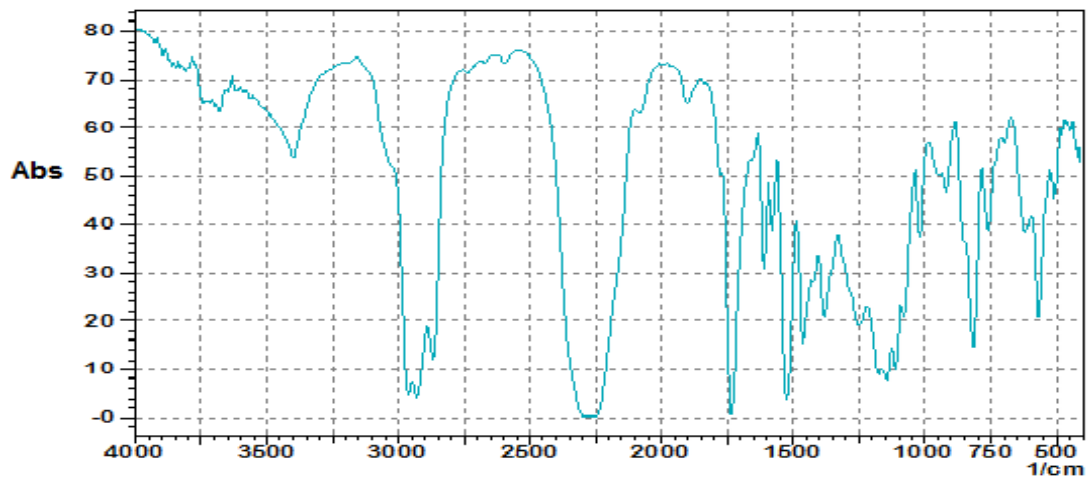


Figure 3. FTIR of (Quickmast 120) polyether polyol.

Figures (4&5) show the FTIR spectrums obtained from the PU foam samples at different (Equivalent ratio) of (isocyanate: polyol) concentrations [(0.5:1),(1:1),(2:1) and (3:1)] for (Local commercial Market polyester and Quickmast 110 polyether). The peak around 2930 cm^{-1} $-\text{CH}_2$ (asymmetric stretching). As part of the main peaks, a signal around $\sim 3310\text{ cm}^{-1}$ can be observed, which is attributed to the N-H stretching vibration from the reaction between isocyanate and polyol to form Urethane linkages ($-\text{NH}-\text{CO}-\text{O}-$) as shown in Figure (4). It doesn't show the absorption bands at 2270 cm^{-1} associated with isocyanate group.

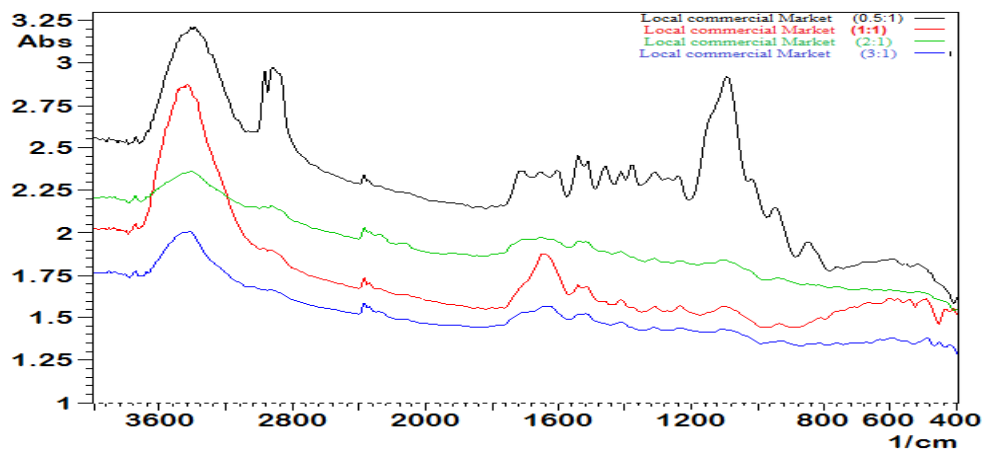


Figure 4. FTIR spectrum of polyurethane foam samples at different equivalent ratio of (isocyanate: polyol) concentrations for (Local commercial Market) polyester polyol.

Figure (5) shows the main peaks of polyurethane samples of (Quickmast 110) polyether polyol. The main peaks at 2862 cm^{-1} and 2938 cm^{-1} are related with $-\text{CH}_2$ stretching, while other mode of $-\text{CH}_2$ vibrations are well-known by the peaks at 1464 , 1418 , 1364 , and 1294 cm^{-1} . The peak owing to the carbonyl (C=O group) stretching bands appeared at 1731 cm^{-1} . The bond at wavelength 3300 cm^{-1} indicates that the hydrogen bonding formed by combining the hydroxyl group (OH) in the polyol with NCO of isocyanate and its IR absorption decreases with increasing isocyanate content. The crosslinking reaction was also monitored by the appearance of the characteristic absorbance of the urethane linkages.

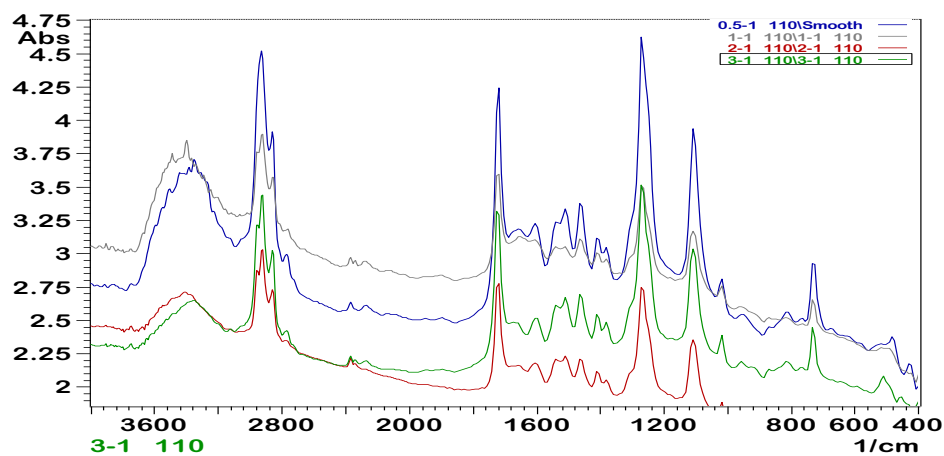


Figure 5. FTIR spectrum of polyurethane foam samples at different (Equivalent ratio) of (isocyanate: polyol) concentrations for (Quickmast 110) polyether polyol.

The absorbance band at lower concentration is higher than the other concentrations since the crosslinking between polyol and isocyanate is not in equilibrium. This is because the OH group in the polyol did not interact equally with the NCO group in the isocyanate (a significant decrease in the intermolecular interaction). This is in agreement with [14].

4.2. Digital microscopy

In this context, the morphology of each polyurethane (PU) foam samples at different (Equivalent ratio) of (polyol: isocyanate) concentrations for (Local commercial Market polyester and Quickmast 110 polyether) polyols was studied using microscopic analysis, and the images collected are shown in Figures (6 & 7) with magnification rate (20x-220x) and image sensor 1.3 Mega Pixel, as detected from the morphology of the polyurethane foams, which involves open cells. Furthermore, it can be detected that the cells at high concentrations are broken and collapsed, while at lower concentrations, specifically (1:1) the general cells structures became further uniform and well-defined, as well as the cells wall becomes thinner. The uniform and thinner cell walls are essential in governing the mechanical characteristic of polyurethane foams this confirmed with [15].

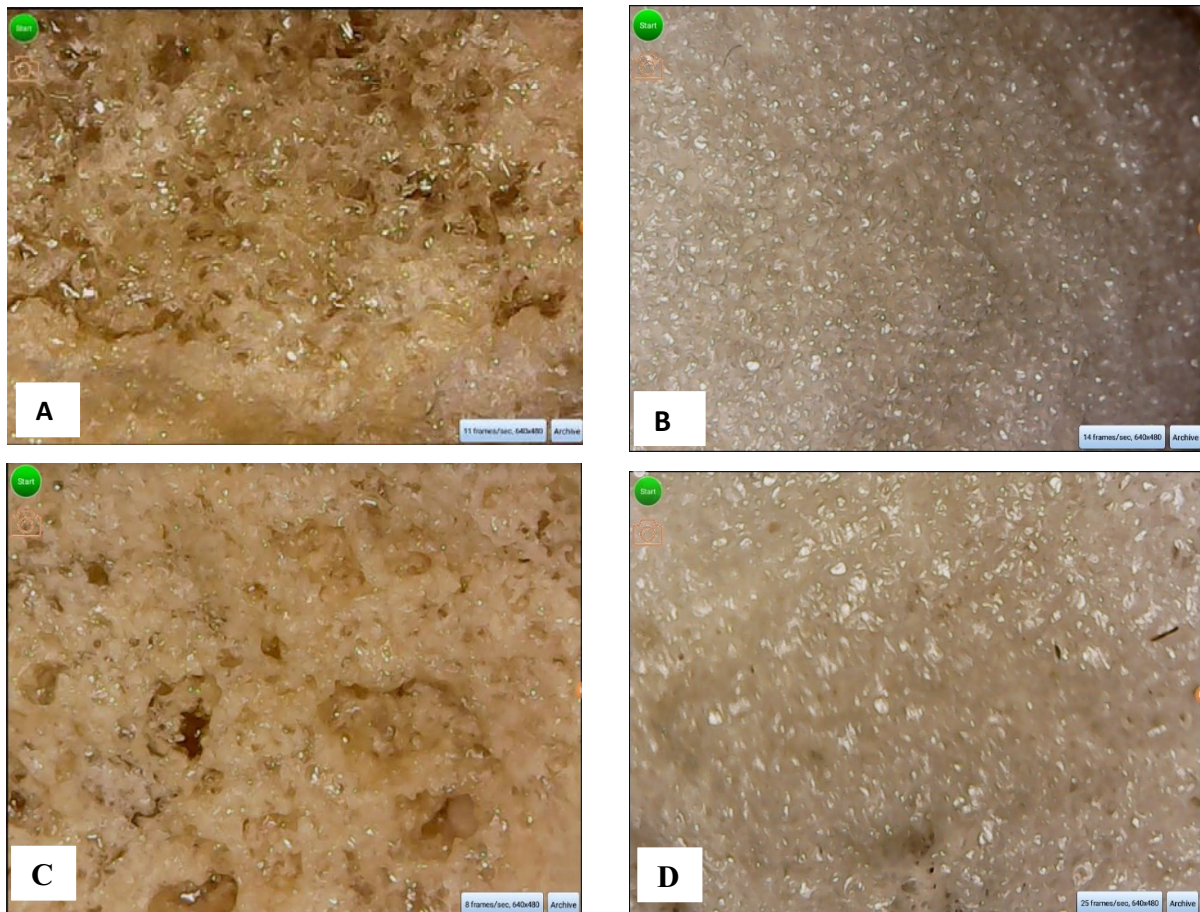


Figure 6. Microstructure for polyurethane samples of (Local commercial Market) polyester at different equivalent ratio of (isocyanate: polyol) concentrations (A) 0.5:1, (B) 1:1, (C) 2:1, and (D) 3:1.

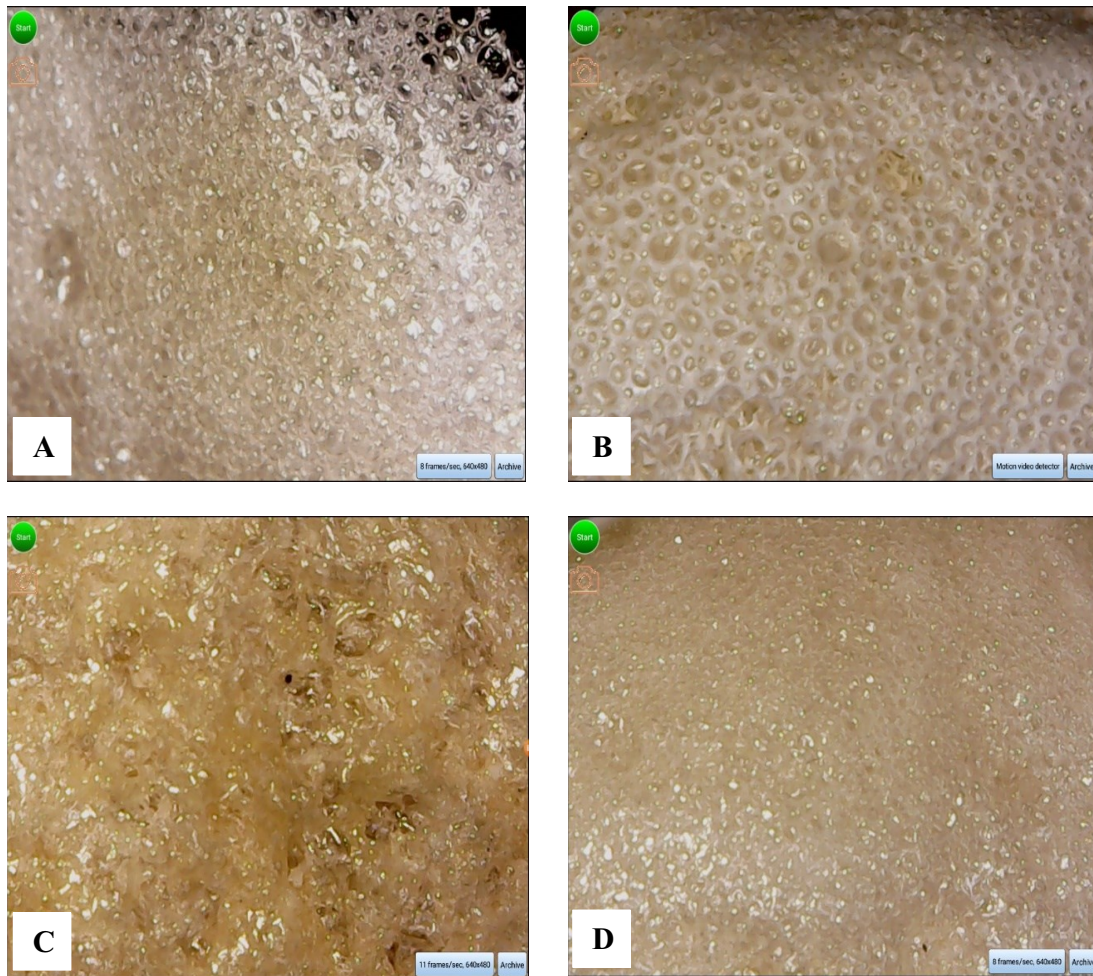


Figure 7. Microstructure for polyurethane samples of (Quickmast 110) polyether at different (Equivalent ratio) of (isocyanate: polyol:) concentrations, (A) 0.5:1, (B) 1:1, (C) 2:1, and (D) 3:1.

Figure (8) Displays the PU foam microstructure of (Quickmast 120) polyether polyurethane samples at different (Equivalent ratio) of (isocyanate: polyol) concentrations with magnification rate (20x-220x) and image sensor 1.3 Mega Pixel. As detected from morphological of the polyurethane foams, the open cells and heterogeneous distribution of cells were dominated. Additionally, it can be seen that the cells size is very big as compared with other type. This unacceptable result does not meet the requirements of the lower limbs prostheses application.

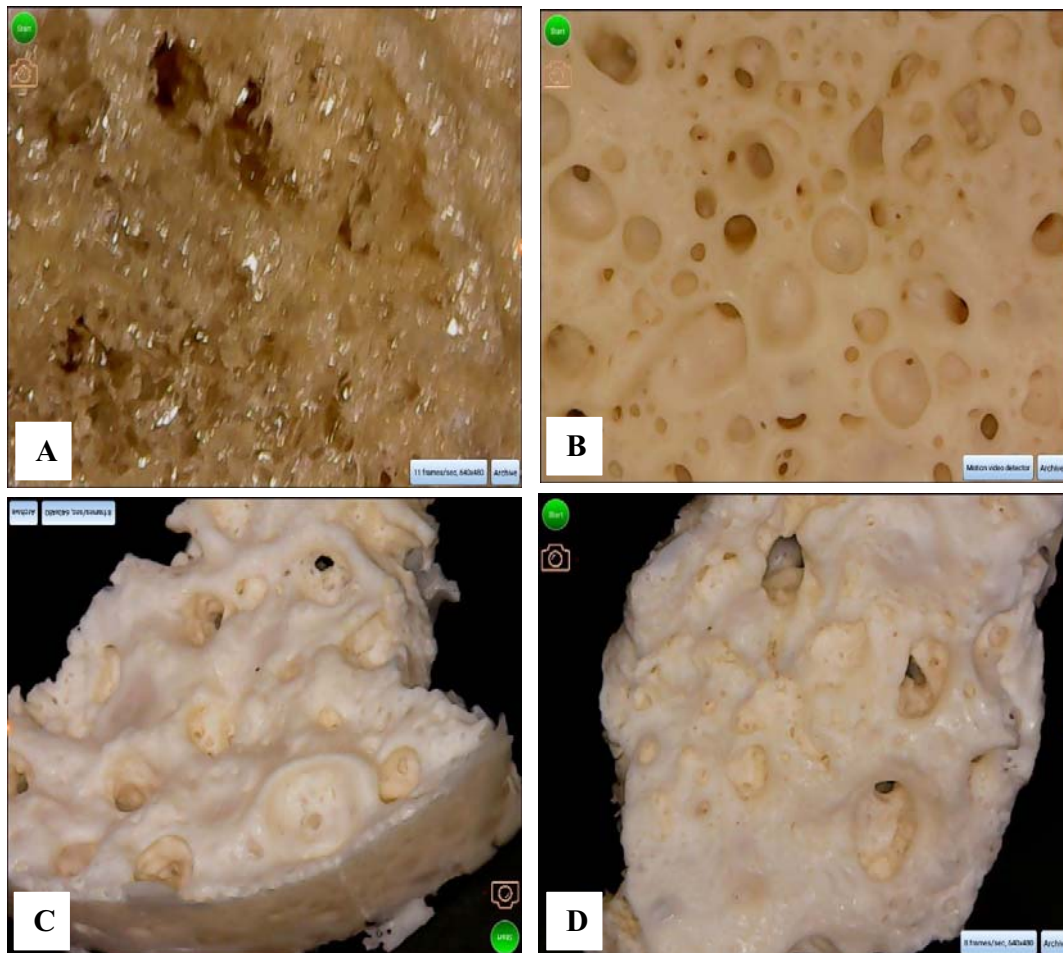


Figure 8. Microstructure for polyurethane samples of (Quickmast 120) polyether at different concentrations of isocyanate: polyol, (A) 0.5:1, (B) 1:1, (C) 2:1, (D) 3:1.

4.3. Rheological properties

The viscosity is an important fluid property in order to study the flow performance of the resin. The distinction is that many of the resin could be sticky solution at 25 °C and provides a sign of the processability of resin. The shear viscosities were also determined for several shear rates in order to investigate flow curve as shown in Figures (9 & 10). It can be seen that at lower shear rate, the viscosity is approximately constant up to the 80 shear rate, after that the viscosity is increasing rapidly up to the shear rate 140 for local commercial market polyester, while for Quickmast 110 the viscosity is approximately constant up to the 33 shear rate, after that the viscosity is irregularly increasing with shear rate as shown in Figure (10). This flow is characterized as dilatant and is in agreement with the [16]. This result shows that the values of viscosity in the similar variety as polyol utilized in preparation of polyurethane foam.

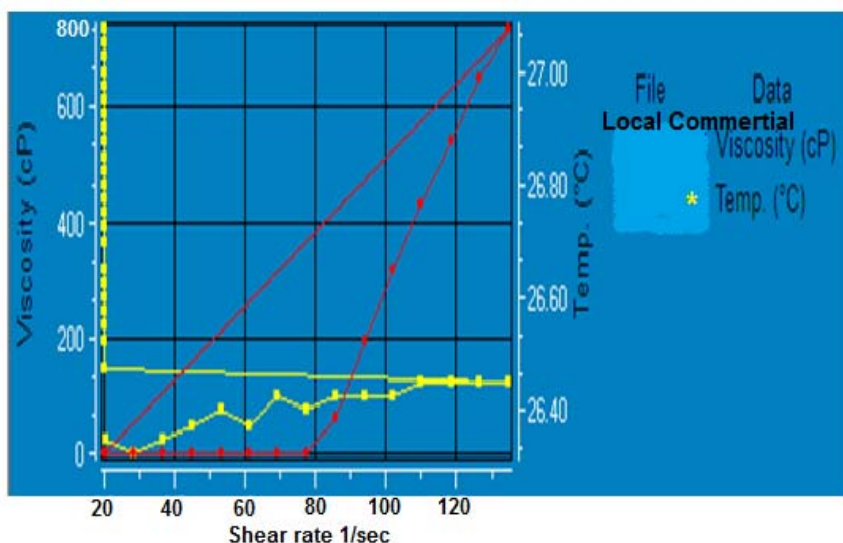


Figure 9. (Local commercial market) polyester polyol shear viscosity a function of the shear rate.

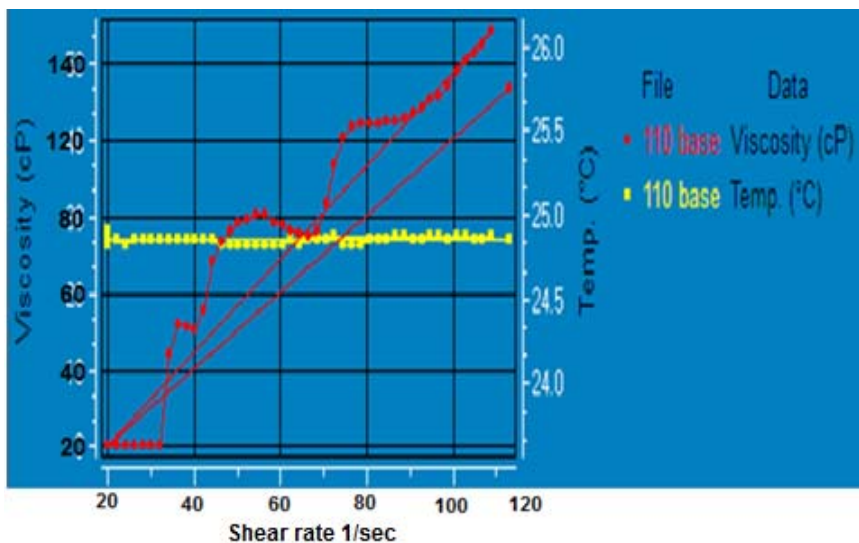


Figure 10. (Quickmast 110) polyester polyol shear viscosity as a function of the shear rate.

Figure (11) Shows the viscosity behavior as a function of shear rate. The results show that the Quickmast 120) polyether polyol exhibits both of shear thickening and shear thinning flow. At very low values of shear rate the viscosity remains constant then it increases (shear thickening) then at 40 (1/sec) shear rate started to decrease given (shear thinning) behavior, this unstable behavior of polyol at different shear rate is responsible for the main morphology (cell size & cell size distribution) and properties of the PU foams.

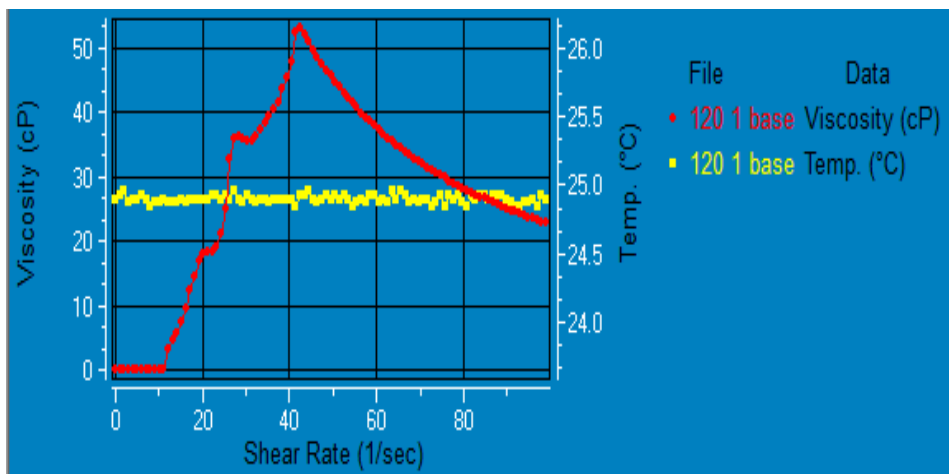


Figure 11. (Quickmast 120) polyether polyol shear viscosity as a function of the shear rate.

Figures (12 & 13) display that the crosslinker (isocyanate) is generally lower in viscosity than resin (polyol). Hence, to improve the mixing efficiency, it will only be useful or necessary to fill crosslinker portion B, due to the highly polar nature of its components.

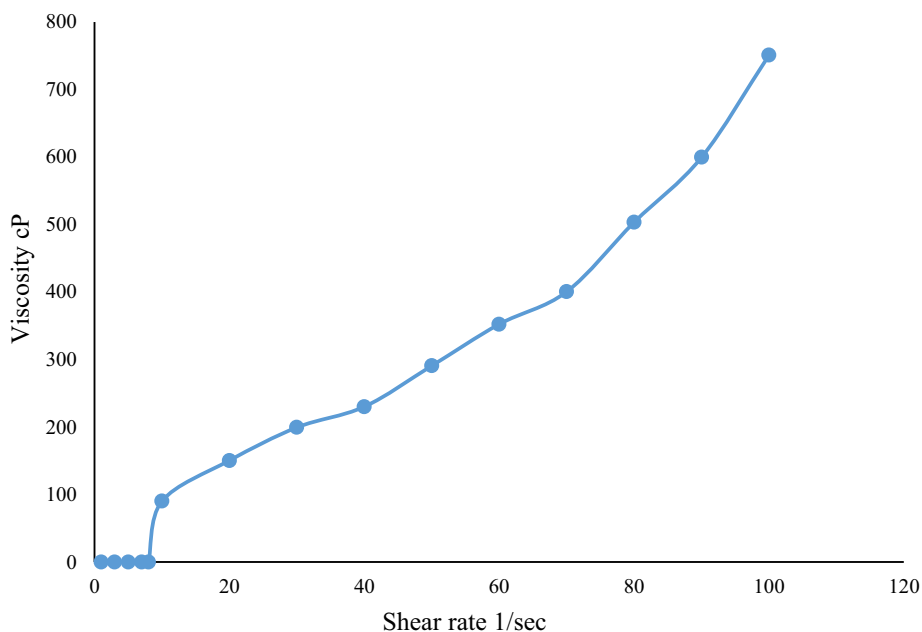


Figure 12. Local commercial market isocyanate shear viscosity as a function of the shear rate.

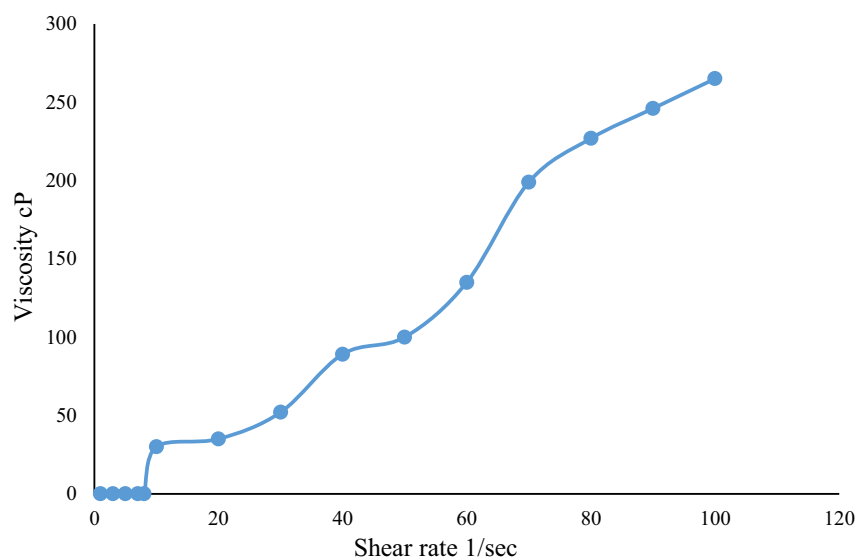


Figure 13. Quickmast 110 isocyanate shear viscosity as a function of the shear rate.

4.4. Mechanical properties

The mechanical properties of PUR foams depend considerably on the parameters of the polymeric matrix composition.

4.4.1. Hardness properties. It is known that the hardness of materials depend on the resistance to penetration at the outer surface. The applications of the foam require well control on the mechanical properties especially hardness. Figures 14 shows the effect of isocyanate concentrations on the hardness of the PU foam for (Local commercial market polyester, Quickmast 110 polyether and Quickmast 120 polyether) polyols. The results show that the hardness increases with increasing the (Equivalent ratio) of (isocyanate: polyol) concentrations. This increase in the hardness is directly related to increasing covalent cross-linking resulting from the complete consumption of isocyanate reactive sites caused by the presence of excess isocyanate groups. This is compatible with the findings of [17, 18]. Control of the hardness is one major issue in the production of flexible polyurethane foam. Reduction of hardness usually is a minor problem. It can be achieved either by the addition of softening additives while keeping the MDI content constant or by reducing the MDI content as it be done in this study.

The Quickmast 120 polyether poly shows higher value of hardness as compared to other types of polyols, and its hardness is very high that is undesirable and unacceptable for the production of highly flexible polymeric foams that are widely used in lower prostheses, which require high flexibility with excellent mechanical properties. In addition, the unstable rheological behavior of this type as shown in Figure (11), also to the high cell size and heterogeneous cell distribution of the final foam as shown in Figure (8) makes it unsuitable for use in the present work.

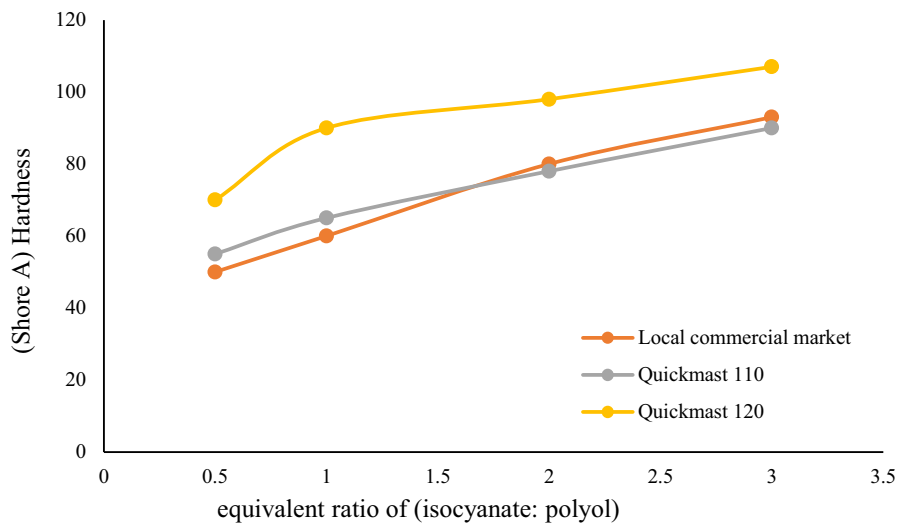


Figure 14. PU foam hardness as a function of the (Equivalent ratio) of (isocyanate: polyol) concentrations for types of polyols.

4.4.2. Tensile properties. The tensile strength and modulus of PU foam sample at different ((Equivalent ratio) of (polyol: isocyanate) concentrations can be shown in Figures (15, and 16) for (Local commercial market polyester and Quickmast 110 polyether). From the result obtained it can be seen that the sample with (Equivalent ratio) of (isocyanate: polyol) of (1:1) has the best quality in terms of tensile strength and modulus of elasticity, in which the tensile strength value is (0.398 MPa) and tensile modulus is (16.32 MPa) for (local commercial market polyester polyol), while for (Quickmast 110 polyether polyol) the tensile strength and tensile modulus is (0.385 MPa and 15.35 MPa) respectively when compared to other foam samples at different (isocyanate: polyol) concentrations, which show less tensile strength and modulus, this is compatible with the results of [9].

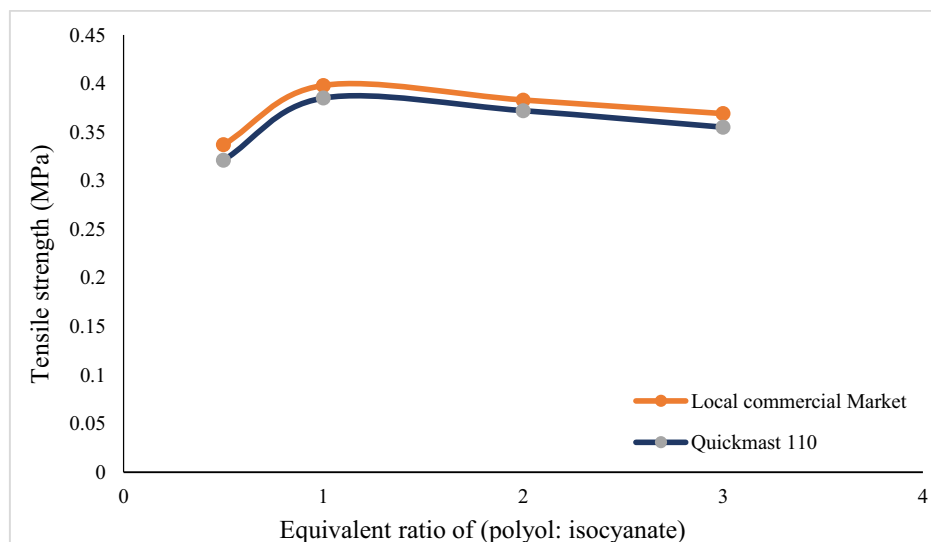


Figure 15. PU foam tensile strength as a function of the (Equivalent ratio) of (isocyanate: polyol) concentrations for different of polyols.

The results of the tensile strength and modulus of the (local commercial market) polyester foams are higher than that of (Quickmast 110) polyether foams because the hydrogen bonding between the ether oxygen and N-H groups is weaker than the interaction between ester carbonyl groups of and N-H groups, resulting in less micro-phase segregation in PU based on polyester. In addition, the viscosity of polyester is higher than of polyether samples and then the formability is better, also morphology is more homogenous and then the cell size is less so that the tensile properties are high. The tensile strength of both local commercial market polyester and Quickmast 110 polyether polyurethanes increases with (Equivalent ratio) of (isocyanate: polyol) concentrations increasing but for high ratio it decreases because the mechanical properties of the PU foams are affected greatly by the polymeric matrix' cross-link density of covalent bond, which increases mainly with the isocyanate content increasing.

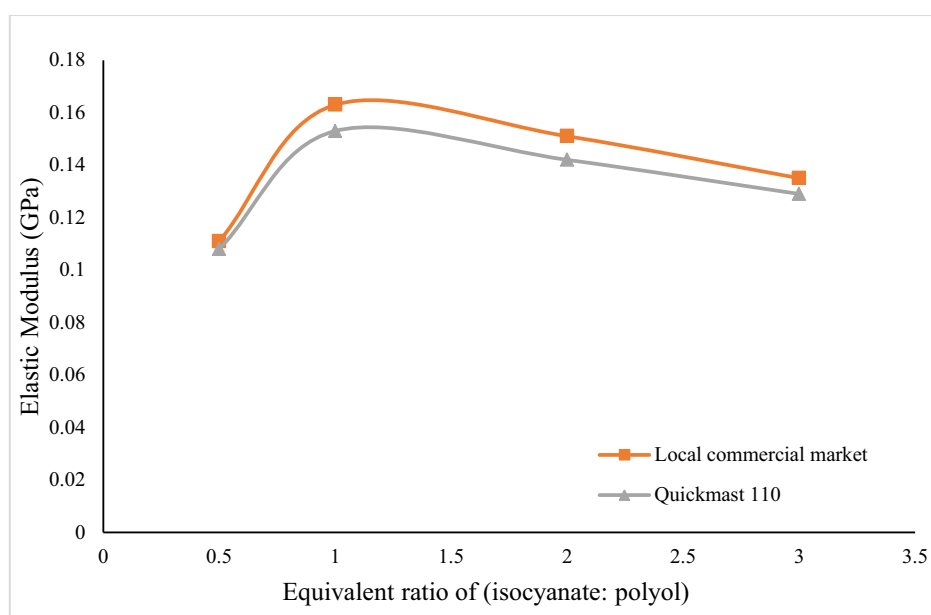


Figure 16. PU foam elastic modulus as a function of the (Equivalent ratio) of (isocyanate: polyol) concentrations for different types of polyols.

4.4.3. Compression properties. Figures (17) illustrates the compressive strength of PU foam sample at different (Equivalent ratio) of (isocyanate: polyol) concentrations for both of (Local commercial market polyester and Quickmast 110 polyether) polyols. The results show that with increasing the (equivalent ratio) of isocyanate at constant polyol content in reactants, the compressive strength of the produced PU foams increases (which is a sign of good and durable quality) and then slightly decreases at higher concentrations of polyol. Foam samples with concentration (1:1) of (isocyanate: polyol) displays higher compressive strength than other foam samples with different isocyanate content. Furthermore, the compressive strength of the (Local Commercial Market) polyester polyurethane foams is bigger than that of (Quickmast 110) polyether polyurethane foams. The compression strength indicates that the foam of higher (equivalent ratio) of isocyanate which indicates that the hardness is very high meaning that the tendency of the foam to return to its original size after compressions is very low compared to the other concentrations, this is in good agreement with the results of [9].

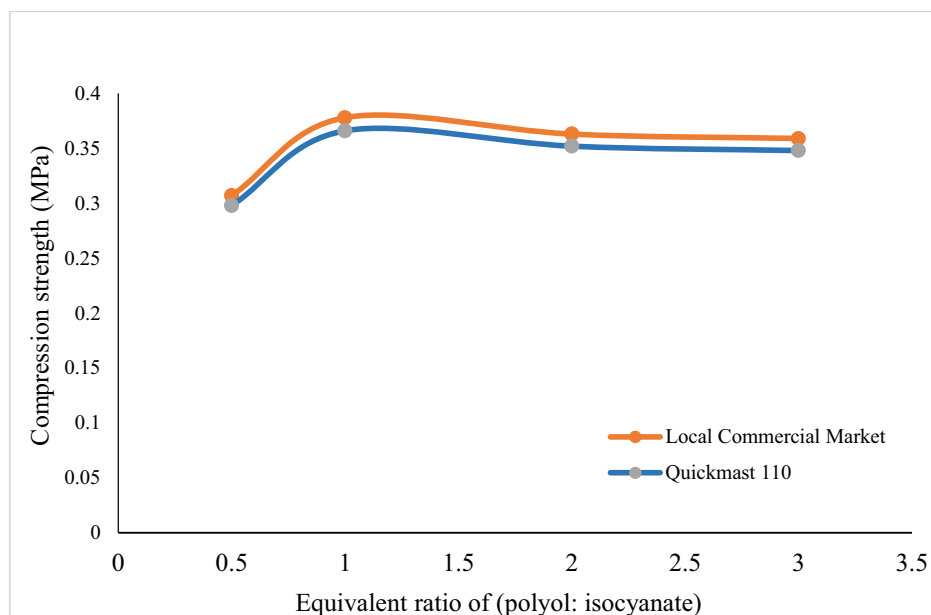


Figure 17. PU foam compression strength as a function of the (Equivalent ratio) of (isocyanate: polyol) concentrations for (Local commercial market) polyester polyol.

5. Conclusion

From this research, it can be concluded that the properties of polyurethane foams depend largely on the type of polyol that is included in the foam formation, as it is noticed that the properties of (local commercial market) polyester foam are better than Quickmast 110 polyether foam. The final properties of the polyurethane foam depend mainly on the concentrations of isocyanate to form urethane linkages. The best morphological, mechanical properties obtained are at (1:1) (isocyanate: polyol) concentrations. Rheological characteristics play an essential role in the foaming process, as noted that the Quickmast 120 polyether polyol had an unstable rheological behaviour and thus the foam formation process was difficult and thus gave relatively large and heterogeneous cell.

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