Synthesis and Biocompatibility of New Contact Lenses Based On Derivatives of 2-Hydroxy Ethyl Meth Acrylate and 2-Ethyl Hexyl Methacrylate

Dr. Mohammed A. Muter

Chemistry Department College of Education University of Al-Qadisiya, Iraq mohammeddw73@gmail.com

Abstract: In the contact lens industry, copolymerization is extensively used in the synthesis of hydrogel materials .Almost all hydrogel materials are copolymers. By polymerizing various combinations of monomers, the physical and chemical properties of a lens can be modified. This work describes the synthesis of new soft contact lenses based on2-hydroxyl ethyl methacrylate (HEMA) and 2-ethylhexy methacrylate (EHMA) monomers by free radical polymerization in the presence of ethylene glycol dimethacrylate (EGDMA) and 1, 6-Hexanediol diacrylate (HDODA) as a crosslinking agent and benzoyl peroxide as an initiator. FTIR was performed to find out the total conversion of HEMA and EHMA monomers into a hydrogel contact lens.

The swelling was measured for all the hydrogels at 34°C, and it found that the hydrophilic hydrogels swelling more than hydrophobic hydrogels. Oxygen permeability (DK) was measured by Morgan & Efron equation, the results show, the hydrophilic hydrogels display higher oxygen permeability towards the cornea more than hydrophobic hydrogels. The amount of proteins adsorbed on the membrane surface of hydrogels contact lenses was calculated, the results show that albumin deposits in higher concentrations on hydrophobic surfaces, with comparison to relatively hydrophilic surfaces. The transparency of all hydrogel was examined using UV-visible spectrophotometer and the results show that all hydrogel contact lenses allow range of light transmittance 57-94.6%, at wavelengths between 400-700 (nm).

Keyword: *Hydrogel, Contact lenses, Oxygen permability, Proteinadsorption, Transmittance measurement, swelling.*

1. INTRODUCTION

Contact lenses are defined as thin curved disks made out of a clear material and can be classified in a number of ways, yet the two main categories are the hard and the soft lenses depending on the material used for their manufacture. A further division of the soft lens category can be made into two sub categories, hydrophobic and hydrophilic sub categories. Thus, the development of contact lens materials takes three specific directions. The first is that hydrogel with high water content, the second is the rigid gas-permeable lenses with enhanced oxygen permeability's. And the third, surface is the modification of silicone elastomer lenses. Such polymeric systems are expected to improve the water content of the contact lenses and the permeability to oxygen as well [Garrett et al. 2002].

Copolymerization in contact lenses' industry is extensively used in the synthesis of materials. Almost all hydrogel materials are copolymers. The physical and chemical properties of a lens can be modified polymerizing various combination of monomers. Also, through varying the ratio of the same two monomers, materials can be created having various water contents, refracture indices, hardness, mechanic strengths and oxygen permeability as well. Many varied hydrogel contact lens materials are available containing 30% to 80 % by weight of water and are based on lightly crosslinked (0.5-1.0)% combination of various monomer including, for example, 2-hydroxyethyl methacrylate (HEMA), acrylamide (AAm), acrylic acid (AA),ethylene glycol dimethacrylate (EGDMA), Poly vinyl alcohols (PVA), chitosan (Cs), ethylhexyl methacrylate (EHMA), poly vinyl pyrrolidone (PVP), Methylacrylate (MA) and Methyl cellulose (MC). Of all poly (HEMA) cross-linked with EGDMA is the basis of many types of soft contact lenses [Pedley

et al. 1980]. A useful property of hydrogel contact lenses is water imbibition of hydrogels because the greater the amount of water content of a lens, the greater the amount of dissolved oxygen in the lens and, in turn, the greater the amount of atmospheric oxygen that can reach the cornea [Refojoet al.1991]. Water imbibition is the ability of the contact lens to absorb water and swell. It is a property that depends on the ratio of hydrophilic to hydrophobic functional groups on the polymer and on the nature of these groups as well [Refojo.1991]. It is one of the most contaminants that is most frequently occurring on contact lenses, and it results from the interaction between the tear film and polymer of a contact lens[Michaudet al. 1998;Merindano et al.1998].Yet the hydrogels used in soft contact lenses and containing AA adsorbs considerably more protein than other gels [Holly1984; Sack et al.1987]. The hydrogel spoilage from tear film is one of the major problems with hydrophilic contact lenses. Such spoilage of contact lenses is because of the different factor such as calcium films, organic plaques and protein films [Bohnertet al.1988]. The important thing for the wearers of contact lenses and for practitioners is transmittance properties of theses contact lenses. And this is not only for ultraviolet radiation (UVR) protective properties, but also for visual performance [Garrettet al.1999; Gallagher et al. 2006].

The present study includes polymerization of 2-HEMA and 2-EHMA by free radical polymerization to produce hydrogel contact lenses and study swelling measured, protein adsorption, oxygen permeability and transmittance for these hydrogel lenses.

2. EXPERIMENTAL

2.1. The Materials

Hydroxylethyl methacrylate (MERCK), Methylacrylate (ALDRICH), Chitosan (ACROS), Ethylhexyl methacrylate (MERCK), acrylic acid (MERCK), Sodium hydroxide (BDH), Acetic acid(BDH), Phosphate buffered saline (HIMEDA), Benzoyl peroxide (HIMEDA), Ethylene glycol dimethacrylate (MERCK), Bovine serum albumin (HIMEDA), Sodium dodecyl Sulfate (HIMEDA) and 1,6 hexandioldiacrylate (ALDRICH).

2.2. Apparatus

(**Oven**)Trivp International Crop. Italy, (**FTIR 8400S**) Fourier Transform infrared spectrophotometer,Shimadzu,Jaban,(**UV-1800PC**),Ultraviolet-visiblespectrophotometer, Shimadaz, Italy, was used to measure transmittance of hydrogel contact lenses.(**UV-1650 PC**) Ultra violet-visible spectrophotometer, Shimadaz, Jaban, was used to the measure of protein adsorption.(**Hotplate stir**)BibbyStrlintd.UK(**PH meter**)Hanna, Romania.

2.3. Polymerization (HEMA)-Based Hydrogels

A (0.015) mole of HEMA with different moles of EGDMA (0.0025, 0.0037, 0.005, 0.006, 0.007) as a crosslinking agent have been polymerized. The polymerization was initiated by using 0.0012 mole benzoyl peroxide dissolved in 5 ml DMF was used an initiator. The mixture was refluxing for 6hrs at 80°C.Nitrogen gas was bubbled throughout solutions about 6hrs. When polymerization has been completed, the hydrogel was removed carefully, and then the hydrogel was dried under vacuum at $(37^{\circ}C)$ for overnight. The dry hydrogel of each was weighed [Ferreira et al. 2006].

2.4. Copolymerization

2.4.1. Copolymerization of (HEMA-co-AAm)

HEMA with AAm by using three different percentage molar ratios (10%, 30%, 50%). When mixed AAm with HEMA,1ml of N,N,N',N'-tetramethlene ethylene diamine (TEMED) was added as an accelerator. Then added 0.0025 moles (EGDMA) as crosslinking agent and 0.0012 moles of BPO dissolved in 5 ml DMF was used an initiator. The mixture was refluxing for 6 hrs at 80°C, nitrogen gas was bubbled throughout the solutions for 6 hrs. When polymerization has been complete, the hydrogels was removed carefully, then the hydrogels were dried in a vacuum oven at $(37^{\circ}C)$ for overnight. The dry hydrogel of each was weighed [Sadeghi 2010].

2.4.2. Copolymerization of (HEMA-co-AA)

The copolymer was synthesized from the reaction of HEMA with AA by using three different percentage molar ratios (10%, 30%, 50%).AA was prepared by naturalization sodium hydroxide

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before use. Then the solutions were added to HEMA monomer. Then added 0.001 moles1,6-Hexanediol diacrylate (HDODA) as crosslinking agent and 0.0012moles of BPO dissolved in 5 ml DMF was used an initiator. The mixture was refluxing for 6 hours at 80°C, nitrogen gas was bubbled throughout the solutions for 6 hrs. When polymerization has been complete, the hydrogels were removed carefully, then the hydrogels was dried in a vacuum oven at (37°C) for overnight. The dry hydrogel of each was weighed. [Renjha et al .2013]

2.5. Terpolymerization with HEMA

The terpolymer was synthesized from reaction of HEMA with the monomers (Cs, MC, EHMA, PVA), using three different percentage molar ratios(40%,40%,20%).Cs or MC was dissolved in acetic acid solution as shown in Table 1.On the other hand (10)gm of PVA was dissolved in 90 ml distillate water. The solution was refluxed at 90°C for 6 hours and kept at room temperature for 24 hours before using. When mixed (Cs, MC, EHMA, PVA), with HEMA, 0.0025 moles (EGDMA) were added as crosslinking agent and 0.0012 moles of BPO dissolved in 5 ml DMF was used an initiator. The mixture was refluxing for 6 hrs at 80°C, nitrogen gas was bubbled throughout the solutions for 6 hrs. When polymerization has been complete, the hydrogels was removed carefully, then the hydrogels was dried in a vacuum oven at (37°C) for overnight. The dry hydrogel of each was weighed Standard formulations for the 3 hydrogels used in this study are listed in Table 2[Bengani et al.2012].

Cs, MC %	Wight (gm)	Vml acetic acid 0.1 (moles)
10%	0.2	15
30%	0.6	30
50%	1	50

Table1. Percentage of Cs, MC in acetic acid

Material	Percer	Percentage weight%			Crosslinking 0.0025 (moles)
HEMA	40	40	40	40	EGDMA
EHMA	20	-	-	-	EGDMA
MC	40	-	-	-	EGDMA
PVA	-	20	-	-	EGDMA
Cs	-	40	-	-	EGDMA

Table2. Different terpolymer used in this study

2.6. Polymerization of (EHMA) Based Hydrogel

A(0.01) moles EHMA with a different concentrations of HDODA (0.001,0.002, 0.003,0.004,0.005) moles as acrosslinking agent was polymerized. The polymerization begins when using (0.0012) moles of benzoyl peroxide was used an initiator. The mixture was refluxing for 6 hrs at 80° C, nitrogen gas bubbled throughout the solutions for 6 hours. When polymerization has been complete, the hydrogel was removed carefully, then the hydrogels was dried in a vacuum oven at (37°C) for overnight. The dry hydrogel of each was weighed [Bengani et al.2012].

2.7. Terpolymerization of EHMA

The terpolymer was synthesized from reaction of EHMA with the monomers PVP and MA by using three different percentage molar ratios (40%,40%,20%) as shown in Table 3.When mixing EHMA with PVP and MA, 0.001 moles (HDODA) were added as crosslinking agent and 0.0012 moles of BPO dissolved in 5 ml DMF was used an initiator. The mixture was refluxing for 6 hrs at 80°C, nitrogen was bubbled throughout the solutions for 6 hrs. When polymerization has been complete, the hydrogels was removed carefully, then the hydrogels was dried in vacuum oven at 37°C for overnight. The dry hydrogel of each was weighed [Bengani et al.2012].

Table3. Terpolymer used EHMA in this study

ſ	Material EHMA MA PVP Crosslinking 0.01(mole)									
	%	40	20	40	HDODA					

2.8. Swelling Measurement

The equilibrium water content in the distillate water was determined by the ratio of the weight of water in the hydrogel to the total weight of the hydrogel at hydration equilibrium. EWC was calculated by using the following equation.

$$EWC = \frac{Ws-Wd}{Ws} \times 100$$

Where Ws and Wdcorrespond to the weight of the swollen sample and dried sample, respectively [Jones 2005].

2.9. Oxygen Permeability

The relationship between EWC and oxygen permeability has been found to be (Morgan &Efron equation):

Where 'EWC' is the Equilibrium water content of the material. The units of Dkare known as Barrer: [Morgan 1998]

2.10.Protein Adsorption

The hydrogel membranes were immersed in phosphate buffer solution (PBS, pH 7.4) 5ml for 24 hrs. After this step soaking the hydrogel membranes in a single protein solution of Bovine serum albumin for 3 hrs at 34 °C the typical surface temperature of the human eye, the concentration of protein solution was 0.5mg/ml. After adsorption the hydrogel membranes were placed gently in to PBS for 5 second to remove excess solution adhering to the hydrogel membranes. The membranes were then inserted into a glass tube containing 1wt% aqueous solution of sodium dodecyl sulfate (SDS) to remove the proteins adsorbed on the membranes. The amount of proteins adsorbed on the membrane surface was calculated from the concentration of proteins in the SDS solution using the Bradford assay reagent kit [Higuchiaet al .2003].

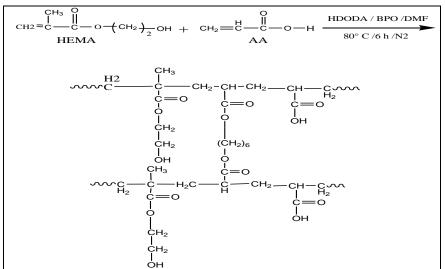
2.11.Transparency Measurement

The transparency of the membrane was examined using UV-visible spectrophotometer, the sample was prepared by solvent evaporation method and immersed in distill water for 24 hours to reach swelling equilibrium. The measurement was performed from 200 to 700 (nm) wavelength at room temperature [Li et al.2011].

3. RESULTS & DISCUSSION

3.1. Synthesis and Characterization

3.1.1. Synthesis and Characterization of Hydrogel (HEMA-co-AA)



Scheme (3-1) Copolymerization and Crosslinking with EGDMA of (HEMA-co-AA)

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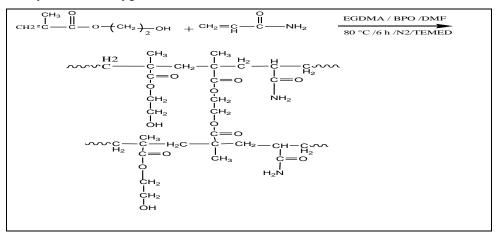
The (HEMA-*co*-AA) was synthesized from the reaction of (HEMA) with (AA) by using different percentage ratios (10%, 30%, 50%) in presence of HDODA as acrosslinking agent and BPO as an initiator by refluxing it with DMF as solvent for 6 hours. The mixture was gently stirred while nitrogen purged through the mixture to remove any dissolved oxygen. This reaction was shown in Scheme 1.

FTIR Spectrum

The FTIR Spectrum of (HEMA-*co*-AA), is shown in Figure 1, which indicates the absorption band at 3433cm⁻¹ to (-OH str group), 3402cm⁻¹ to(COOH str group)2938cm⁻¹,2829cm⁻¹ to(C-Hstr aliphatic polymer backbone), 1750cm⁻¹ (C=O str ester group),1149cm⁻¹ to (C-O-C str) and 1082 cm⁻¹to(-C-O of C-OH str)[Silverstien et al.2011; Pretsch et al .2009; Pavia et al.2009;Rao.1963].

3.2. Synthesis and Characterization of Hydrogel (HEMA-co-AAm)

The (HEMA-*co*-AAm) was synthesized from the reaction of (HEMA) with (AAm) by using different percentage ratios (10%,30%,50%) in the presence of EGDMA as a crosslinking agent, BPO as an initiator, and 1ml of TEMED was added as accelerator by refluxing it with DMF as solvent for 6 hrs. The mixture was gently stirred while nitrogen was purged through the mixture to remove any dissolved oxygen. This reaction was shown in Scheme 2.

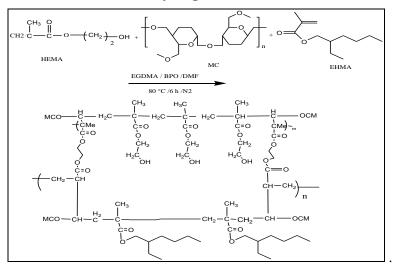


Scheme (3-2) Copolymerization and Crosslinking with EGDMA of (HEMA-co-AAm)

FTIR Spectrum

FTIR Spectrum of (HEMA-*co*-AAm), is shown in Figure 2 , which indicates the absorption band at; 3355cm^{-1} to (-OH str group), 3311cm^{-1} to (N-H str group), 2947cm^{-1} , 2885cm^{-1} to (C-H str group of polymer backbone), 1720cm^{-1} to (ester group, C=O str group), 1674 cm^{-1} to (N-H-C=O)1380 cm⁻¹ to (C-N), 1157 cm⁻¹ to (C-O-C str group) and 1080 cm⁻¹ to (-C-O of C-OH str)[Silverstien et al.2011; Pretsch et al.2009; Pavia et al.2009; Rao.1963].

3.3. Synthesis and Characterization of Hydrogel (HEMA-co-MC-co- EHMA)



Scheme (3-3) Copolymerization and Crosslinking with EGDMA of (HEMA-co-MC-co-EHMA)

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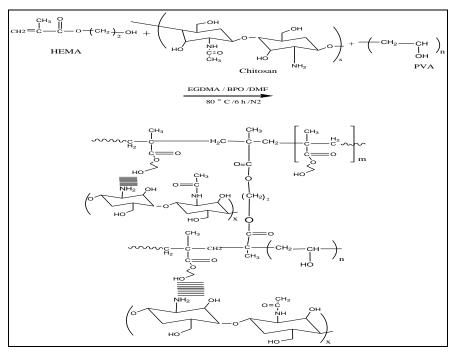
The (HEMA-*co*-MC-*co*-EHMA) was synthesized from the reaction of HEMA with MC and EHMA in the presence of EGDMA as a crosslinking agent and BPO as an initiator by refluxing it with DMF as solvent for 6 hours. The mixture was gently stirred nitrogen was purged through the mixture to remove any dissolved oxygen. This reaction was shown in Scheme 3.

FTIR Spectrum

The FTIR Spectrum of (HEMA-*co*-MC-co-EHMA), is shown in Figure 3 ,which indicated absorption band; The peak in the range of 3440 cm⁻¹ is very broad to hydroxyl group (-OH str), 2923cm⁻¹ ,2854cm⁻¹ to (C-H str of polymer backbone),1728 cm⁻¹ to (ester group, C=O str),1149cm⁻¹ ,1072 cm⁻¹ to (C-O-C str) and 1026 cm⁻¹ to (-C-O of C-OH str).[Silverstien et al.2011; Pretsch et al .2009; Pavia et al.2009; Rao.1963]

3.4. Synthesis and Characterization of Hydrogel (HEMA-co-Cs-co-PVA)

The (HEMA-*co*-Cs-co-PVA) was synthesized from the reaction of (HEMA) with(Cs) and PVA by using different percentage ratios(10%,30%,50%) in the presence of EGDMA as a crosslinking agent and BPO as an initiator by refluxing it with DMF as solvent for 6 hours. The mixture was gently stirred while nitrogen purged through the mixture to remove any dissolved oxygen. This reaction was shown in Scheme 4.



Scheme (3-4) Copolymerization and Crosslinking with EGDMA of (HEMA-co-Cs-co-PVA)

FTIR Spectrum

The FTIR Spectrum of (HEMA-*co*-Cs-co-PVA), is shown in Figure 4, which indicated absorption band at 3379cm⁻¹ to (-OH str group), 3352cm⁻¹ to (N-H str), 2931cm⁻¹, 2885 cm⁻¹ to (C-H str of polymer backbone),1720 cm⁻¹ to (ester group, C=O str), 1658cm⁻¹ to (N-H-C=O), 1072 cm⁻¹ to (C-O-C str),1018 cm⁻¹to (-C-O of C-OH str) and 1157cm⁻¹ to (C-N str)[Silverstien et al.2011; Pretsch et al .2009; Pavia et al.2009; Rao.1963].

3.5. Synthesis and Characterization of Hydrogel (EHMA-co-PVP-co-MA)

The (EHMA-*co*-PVP-*co*-MA) was synthesized from the reaction of (EHMA) with (PVP) and MA in the presence of HDODA as a crosslinking agent and BPO as an initiator by refluxing it with DMF as solvent for 6 hours. The mixture was gently stirred while nitrogen purged through the mixture to remove any dissolved oxygen. This reaction was shown in Scheme 5.

FTIR Spectrum

The FTIR Spectrum of (EHMA-*co*-PVP-*co*-MA), is shown in Figure 5 , which indicates the absorption band; 2947cm⁻¹,2862cm⁻¹to (C-H str of polymer backbone),1735 cm⁻¹ to (ester group,

 $\overline{C=O \text{ str}}$, the absorption band 1450 cm⁻¹, 1388 cm⁻¹ were assigned to the characteristic vibration of the pyrrolidone ring, 1164 cm⁻¹ to (C-O-C str) and 1064 cm⁻¹ to (-C-O of C-OH str)[Silverstien et al.2011; Pretsch et al.2009; Pavia et al.2009; Rao.1963].

3.6. Water Content

3.6.1. Effect of Acrylic acid on Water Content of Hydrogel HEMA

In order to increase the water content and the oxygen permeability of contact lenses the polar monomer, acrylic acid (AA) and 2-hydroxyethyl methacrylate (HEMA) are polymerized into hydrogels. Hydrogels (AA-co-HEMA) have been extensively studied in the biomedical and pharmaceutical fields for a variety of applications including soft contact lenses. The EWC is the most significant single property of the gel since it is the water held within the polymer substrate that gives hydrogels their unique properties. When added AA to a soft lens polymer formulation, it results in a soft lens with ionized groups (negatively charged) within the polymer matrix, allowing the lens to absorb more water. The conversion of the carboxyl group (CO_2H) to the more hydrophilic ionized form (the carboxyl ate anion, CO_2) produces an increase in water content[Castillo et al.1986].This is commonly achieved by treating AA with sodium hydroxide, the carboxylic acid groups are neutralized, as illustrated in the following equation:

$R-CO_2H + NaOH \rightarrow R-CO_2Na + H_2O$

Figure 6: Equilibrium Water Content (EWC) for Different Ratios of AA (10%, 30%, 50%) :

3.6.2. Effect of Acrylamide on the Water Content of Hydrogel HEMA

The use of Acryl amide as a co-polymer with 2-HEMA in order to increase the water content of hydrogels contact lenses which provide hydrogen bonding with water molecules in the site of amine groups[Thakur et al.2011]Figure 7: Equilibrium Water Content (EWC) for Different Ratios of AAm (10%,30%,50%).

3.6.3. Effect of Terpolymer on Water Content

Chitosan, poly vinyl alcohols and Methyl cellulose, are hydrophilic polymers and used in many biomedical applications such as contact lenses because of non toxic and higher water content, which contain many hydroxyl groups in both polymers that make hydrogen bonding with water molecules, so increase the water content of hydrogels 2-HEMA, on other hand ethylhexyl methacrylate and methylacrylate are hydrophobic monomers which reduce the water content because of content methyl groups which decrease the water content of hydrogels contact lenses[Moradi et al.2003]. Figure 8: Equilibrium Water content (EWC) for different ratios of Terpolymer (HEME-co-PVA-co-Cs),(HEMA-co-EHMA-co-MC)and(EHMA-co-PVP-MA)

3.7. Protein Adsorption

During contact lens wear, tear film components such as lipids, mucines and proteins tend to deposit on and within the lens material and may cause discomfort, reduced vision and inflammatory reactions, The concentration of albumin in the tear film is relatively low compared to the concentration in blood serum, but this value increases markedly under various conditions, including when the eye is closed, during contact lens wear and in various dry eye states [Tigheet al.2006].

Synthesized contact lens materials with various concentrations of relatively hydrophobic (MA, EHMA) and hydrophilic (HEMA, AA, Cs, MC, PVA, AAm) components reported that increasing levels of hydrophilic components decreased albumin deposition. Concentrations on hydrophobic surfaces, as compared to relatively hydrophilic surfaces discern electrostatic attraction has a strong impact on albumin deposition. The negatively charged albumin is more likely to deposit on neutral or positively charged substrates than on materials with a net negative charge.Figure9 show the effect of albumin deposition of lens material. Where 1-(HEMA-co-AA), 2-(EHMA-co-AAm), 3-(HEMA-co-PVA-co-Cs), 4-(HEMA-co-EHMA-co-MC), 5-(EHMA -co-PVP -co-MA)

3.8. Transmittance Measurement

The synthesized polymer must be transparent in order to be suitable for contact lenses. The UVR spectrum is subdivided into three bands: UVC (100–280 (nm), UVB (280–315 (nm) and UVA

(315–400 (nm).The results show that all contact lenses tested allowed relatively uniform range of transmittance of approximately 57-95.6%, at wavelengths between 400-700(nm) and below 240 (nm)will be less than 1% transmittance for all contact lenses[Bergmanson et.al.1997]. The percentages of light transmission at 290(nm) for terpolymer (HEMA-*co*-MC-*co*-EHMA),(HEMA-*co*-PVA-*co*-Cs) and (EHMA-co-MA-co-PVP) are (13.8,11.7 and 27.2)% respectively a higher than copolymer (HEMA-*co*-AA) and (HEMA-*co*-AAm) are (6.1,8.3)% respectively. The percentage of light transmission at 400 (nm) for terpolymer (HEMA-*co*-MC-*co*-EHMA),(HEMA-*co*-PVA-*co*-Cs) and (EHMA-co-MA-co-PVP) give higher light transmittance(73, 82.3 and 94.2)% than copolymer give the light transmittance lower than terpolymer (HEMA-*co*-AA), (HEMA-*co*-AAm) (57,69)% respectively.Figure10: Transmittance of Copolymer and Terpolymer in the visible light wave range of 400-700 (nm).

3.9. Oxygen Permeability

The Oxygen permeability increase logarithmically with increase water content. It is essentially governed by EWC in conventional hydrogels. This occurs since oxygen is able to pass through the water rather than through the material itself. The relationship between EWC and oxygen permeability has been found to be (Morgan &Efron equation)[Morgan et.al.1998]. **Dk=1.67e** ^{0.0397EWC}. Figure 11: Show Oxygen Permeability of Lens Material.Where1-(HEMA-co-AA),2-(HEMA-co-AAm), 3-(HEMA-co-Cs-co-PVA), 4-(HEMA-co-MC-co-EHMA), 5-(EHMA-co-MA-co-PVP)

4. CONCLUSION

The results show the equilibrium water content increases with increasing hydrophilic groups in hydrogels contact lenses. The oxygen permeability (Dk) increases logarithmically with increasing water content of hydrogel contact lenses which increases permeability of oxygen and carbon dioxide to promote normal corneal metabolism. Protein adsorption of hydrogel contact lenses shows that albumin deposits in higher concentrations on hydrophobic surfaces as compared to relatively hydrophilic surfaces. The transmittance of light (%T) was studied by UV Spectrophotometer, the transmittance of hydrogels contact lenses shows that all hydrogel contact lenses exhibited light transparency in the range 57-94.6 %.

ACKNOWLEDGMENTS

The authors gratefully acknowledge partial support of this work of Chemistry University of Qadisiyah.

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