표준인증안전학회지(2019. 6)
제9권 제2호 p.55-65.

http://www.standards-standardization.org
https://doi.org/10.34139/JSCS.2019.9.2.55

Standardization of Wettability Evaluation for Ophthalmic Hydrogel Lenses

Shin, Su-Mi* · Sung, A-Young**

The purpose of this study was to analyze the need for standardization of wettability, which is one of the physical properties of contact lenses. This study compared the physical properties of lenses fabricated by adding materials with different wettability levels to basic hydrogel materials to analyze the necessity of standardized specifications for the wettability of lenses. To fabricate lenses, HEMA (2-hydroxyethyl methacrylate), ethylene glycol dimethacrylate (EGDMA, a crosslinking agent), and azobisisobutyronitrile (AIBN, an initiator) were copolymerized by adding N-vinyl-2-pyrrolidone (NVP) and methacrylic acid (MA), which are known as hydrophilic substances, as well as methyl methacrylate (MMA) and butyl methacrylate (BMA), which are known as hydrophobic substances. The refractive index, water content, contact angle, atomic force microscope (AFM), and spectral transmittance were measured to evaluate the basic properties of the fabricated lenses. The results showed that the water content of the hydrogel lens with MMA and BMA decreased by 14 and 27%, respectively, and the water content of the hydrogel lens with NVP and MA increased by about 5 and 28%, respectively, compared to REF. In the case of wettability, that of the hydrogel lens with MMA and BMA decreased by about 6%, and that of the hydrogel lens with NVP and MA increased by 1 and 23%, respectively, compared to REF. Except for the hydrogel lens with MA added, the changes were minimal. Wettability is a major factor affecting the wearing comfort of contact lenses, but there are currently no standardized specifications for it. In addition, the result of the water content in the standardized specifications was not proportional to the wettability result. Therefore, it is necessary to accurately evaluate the performance of the contact lens by standardizing the wettability measurement method.

Key Words: standardized specifications, wettability, water content, contact lens, surface roughness

* Stud. Su-Mi Shin, Dept. of Optometry & Vision Science, Daegu Catholic Univ. (soomi8712@naver.com)
** Prof. A-Young Sung, Dept. of Optometry & Vision Science, Daegu Catholic Univ. (say123sg@hanmail.net), Corresponding Author

Ⅰ. Introduction

The scope of application, test method, test equipment, and scope of approval for contact lenses are based on domestic and overseas test standards such as the IEC standards and KS based on hard/soft contact lenses for MFDS medical device approval. The basic physical properties of contact lenses include the refractive index, water content, spectral transmittance, tensile strength, and wettability. Among these, the factors affecting the wearing comfort and the fitting of the lens are the water content and wettability.(Masnick, K.B., Holden, B.A., 1972; Tranoudis, I. and Efron, N., 2004; Naduvilath, T., Papas, E.B. and Lazon de la Jara, P., 2016) Water content is the percentage of moisture in the weight of the contact lens in the state of hydration. It can be classified with the gravimetric or refractive index method, and the error range should be within ±2%.(Fatt, I. Chaston, J., 1982; Kim, T.H., Ye, K.H. and Sung, A.Y., 2008) The higher the water content is, the greater the flexibility, wearing comfort, and oxygen transmissibility, while the strength and refractive index of the lens material decreases and the deposit adheres well. The low-water-content lenses are easy to handle due to their relatively high strength, and can be made to be thin lenses due to their high refractive index, but they have the disadvantages of low oxygen transmissibility and poor flexibility. Wettability refers to the degree to which liquid spreads on a solid surface when it is dropped thereon, and it represents the degree of wetting of the contact lens surface with tears, which is generally evaluated by measuring the contact angle.(French, K., 2005; Read, M.L., Morgan, P.B., Kelly, J.M. and Maldonado-Codina, C., 2011) A large contact angle means that the wettability is low and the surface is hydrophobic. On the other hand, a small contact angle means that the wettability is high and the surface is hydrophilic. Therefore, the physical properties such as hydrophilicity and hydrophobicity of the solid material can be evaluated by using the contact angle. There are currently various methods for this, classified mainly into static and dynamic measurement methods like the sessile and captive drop methods. Generally, the sessile drop method, a static measurement method, is widely used.(Lorentz, H., Rogers, R. and Jones, L., 2007; Lee, M.J., Lee, H.M. and Sung, A.Y., 2019; Lee, M.J., Shin S.M. and Sung, A.Y., 2019) Many studies have been conducted of late on the methods for increasing the wettability while minimizing the water content, due to the disadvantages of the high-water-content lens.(Lee, C.W., Jeong, H.W., Kim, H.J. and Ryu, G.C., 2017; Lee, M.J., Sung, A.Y. and Kim, T.H., 2014) Wettability has a great role in reducing the repulsion to wearing contact lenses, by improving the wearing comfort.(Kim, H.J., Kim, S.H. and Kim, J.M, 2011) Various physical properties of contact lenses have been standardized of late, including the spectral transmittance, water content, oxygen transmissibility, and refractive index, but the wettability has not been standardized. The current contact lens manufacturing companies, however, do not have actual standardized specifications, so they do not accurately label the wettability performance. Related regulations should immediately be established so as to provide a standardized format. In this study, precise criteria for the contact angle were established among the physical properties of contact lenses, and the performance and physical properties of contact lenses were accurately evaluated, and the criteria for the consumers were presented. In addition, the surface shape and roughness of the contact lenses were checked using an atomic force microscope (AFM), and the correlations with the wettability and tendency were analyzed.

Ⅱ. Experiment

1. Materials and polymerization

In this experiment, having the main material of the hydrogel lens, HEMA (2-hydroxyethyl methacrylate), as a base, the hydrophobic substances MMA (methyl methacrylate) and BMA (butyl methacrylate) and the hydrophilic substances NVP (N-vinyl-2-pyrrolidone) and MA (methacrylic acid) were used to fabricate contact lenses. Additionally, a crosslinking agent, EGDMA (ethylene glycol dimethacrylate), and an initiator, AIBN (azobisisobutyronitrile), were used. The structure of the additives that were used in this experiment is presented in Fig. 1.

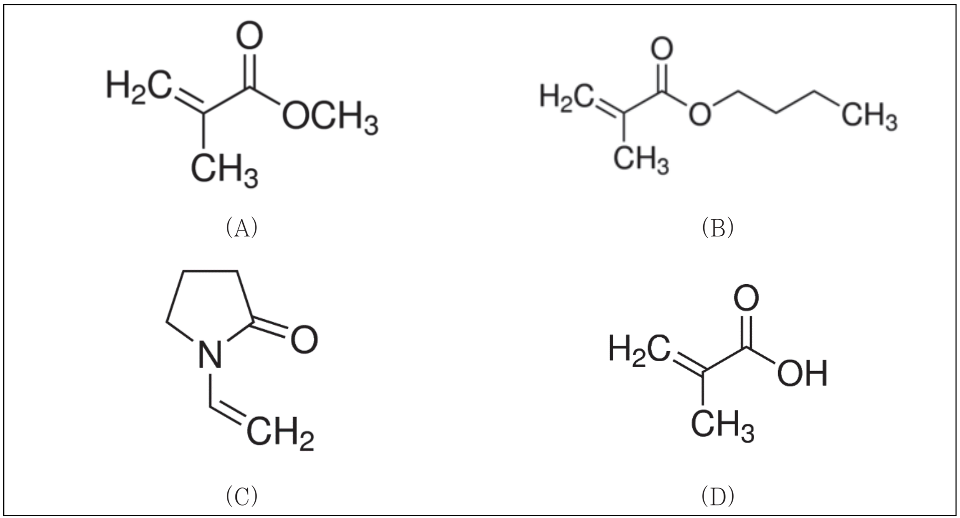
With HEMA, EGDMA, and AIBN as a basic combination, MMA, BMA, NVP, and MA were added, respectively, and then the mixtures were stirred for 60 minutes and heat-treated for another 60 minutes at 100℃ using an oven (Sejong, SJ-201D, South Korea), to copolymerize them. The basic combination without an additive was set to REF, the additive with a hydrophobic nature was set to group 1, and the additive with a hydrophilic nature was set to group 2. The groups were named “1-M,” “1-B,” “2-N,” and “2-A.” The mixing ratio of the hydrogel contact lens that was used in the experiment is presented in Table 1.

2. Physical Property Measurement and Analysis Method

1) Spectral transmittance

The spectral transmittance was measured using an Agilent Cary 60 UV-vis spectrophotometer and based on ISO 8599:1994 (Optics and optical instruments - Contact lenses -

〈Fig. 1〉 Chemical structures of additives



(A) Methyl Methacrylate (B) Butyl Methacrylate (C) N-vinyl-2-pyrrolidone (D) Methacrylic acid

〈Table 1〉 Percent compositions of samples (Unit : wt%)

Sample	HEMA	EGDMA	AIBN	MMA	BMA	NVP	MA	Total
REF	99.30	0.50	0.20	-	-	-	-	100.00
1-M	90.28	0.45	0.18	9.09	-	-	-	100.00
1-B	90.28	0.45	0.18	-	9.09	-	-	100.00
2-N	90.28	0.45	0.18	-	-	9.09	-	100.00
2-A	90.28	0.45	0.18	-	-	-	9.09	100.00

Determination of the spectral and luminous transmittance).

2) Refractive index

To measure the refractive index, an ABBE refractometer (ATAGO NAR 1T, Japan) was used based on ISO 18369-4:2006 (Ophthalmic optic-Contact lenses-Part 4: Physicochemical properties of contact lens materials, 4.5. Refractive index). It was measured after removing the moisture on the surface of the lens with wipe paper that was less prone to lint and was not torn easily.

3) Water content

The water content was measured with the gravimetric method based on ISO 18369-4: 2006 (Ophthalmic optic-Contact lenses-Part 4: Physicochemical properties of contact lens materials, 4.6. water content). The test samples were measured using an electronic balance (Ohaus, PAG 214C, USA).

4) Contact angle

The contact angle was measured with the sessile drop method by flattening the fabricated contact lenses using DSA30 (Kruss GMBH, Germany) and dropping pure distilled water on the surface of each sample. Also, the surface of the hydrated lens was evaluated after appropriately removing the moisture using wipe paper.

5) AFM

AFM was used to study the molecular composition or various physical properties of the local surfaces of the samples. The surface analysis of the fabricated contact lenses was conducted using XE-100, the AFM of Park Systems.

Ⅲ. Results and Discussion

1. Status of the standardized specifications of the relevant areas

The Korean Industrial Standards (KS) of the relevant areas include KS B ISO 8320 (2014), KS B ISO 8321-2 (2013), KS A ISO 5725, and KS B ISO 11978. Also, the status of the contact-lens-related International Organization for Standardization (ISO) includes ISO 18369-1:2006 Ophthalmic optics - contact lenses - Part 1: Vocabulary, classification system and recommendations for labelling specifications, ISO 18369-2:2006 Ophthalmic optics - contact lenses - Part 2: Tolerances, ISO 18369-3:2006 Ophthalmic optics - contact lenses - Part 3: Measurement methods and ISO 18369-4:2006 Optics and optical — Contact lenses— Part 4: Physicochemical properties of contact lens materials, etc.

In the current standardization provisions, there are 19 standards, including the contact lens's shape and appearance, diameter, thickness, radius of curvature, vertex refractive power, cylindrical refractive power and circumference axis, visible light transmittance, ultraviolet light transmittance, water content, oxygen transmissibility, amount of extractables, extractables test, eye irritation test, and sterility test. There is no standard, however, for the wettability of contact lenses. Therefore, it is necessary to establish related regulations to provide an accurate and standardized format of the performance and physical properties for the wettability-related specifications.

2. Spectral Transmittance

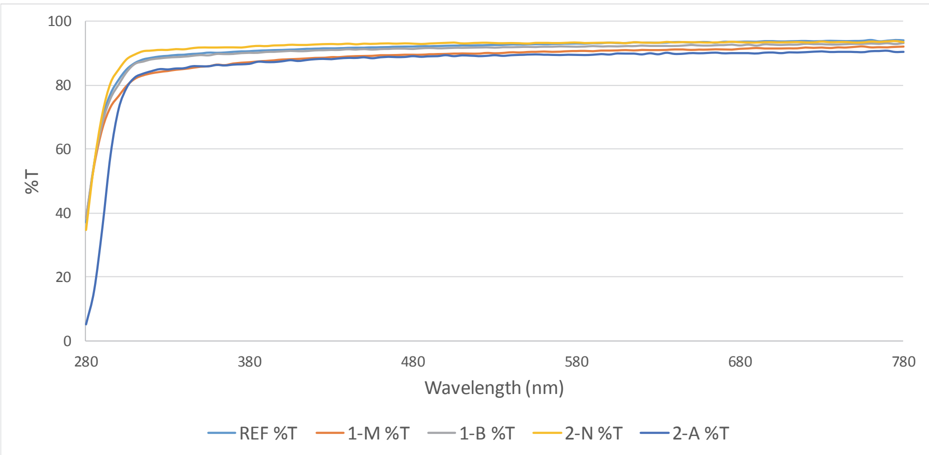
For the measurement results of the spectral transmittance of the fabricated lenses, the transmittance of the visible-light region was about 90% in all the samples, and generally transparent hydrogel contact lenses were made. In addition, the hydrogel lenses with 2-A showed an about 30% reduction in the UV-B region compared to REF, somewhat blocking UV rays. Fig. 2 shows the spectral transmittance according to the additives.

3. Refractive Index and Water Content

The refractive index of the fabricated contact lens was determined to be 1.4333 for REF without an additive. It was 1.4448 for hydrophobic 1-M, 1.4534 for hydrophobic 1-B, 1.4304 for hydrophilic 2-N, and 1.3972 for hydrophilic 2-M. Compared to REF, the hydrogel lens with hydrophobic BMA increased the refractive index compared to MMA, and the hydrogel lens with hydrophilic MA showed a lower refractive index than that with NVP.

The water content was obtained after measuring the wet and dry weights of the fabricated contact lenses. The water content was determined to be 38.53% for REF, 33.20% for 1-M, 28.07% for 1-B, 40.56% for 2-N, and 53.52% for 2-A. The addition of BMA decreased the water content more than the addition of MMA did, and the addition of MA increased the water content more than the addition of NVP did, indicating a general change that was inversely proportional to that in the refractive index.(Anthony, P. and Lynne, S., 2006) It is considered that the structure of BMA is more hydrophobic than that of MMA because the carbon chain is longer than that of MMA, and BMA has a slightly lower water content than MMA.(Ye, K.H., Kim, T.H. and Sung, A.Y., 2008) Moreover, between NVP and MA, which are used as materials for the typical-water-content contact lenses, the addition of MA was found to have

〈Fig. 2〉 Refractive Index and water content of samples



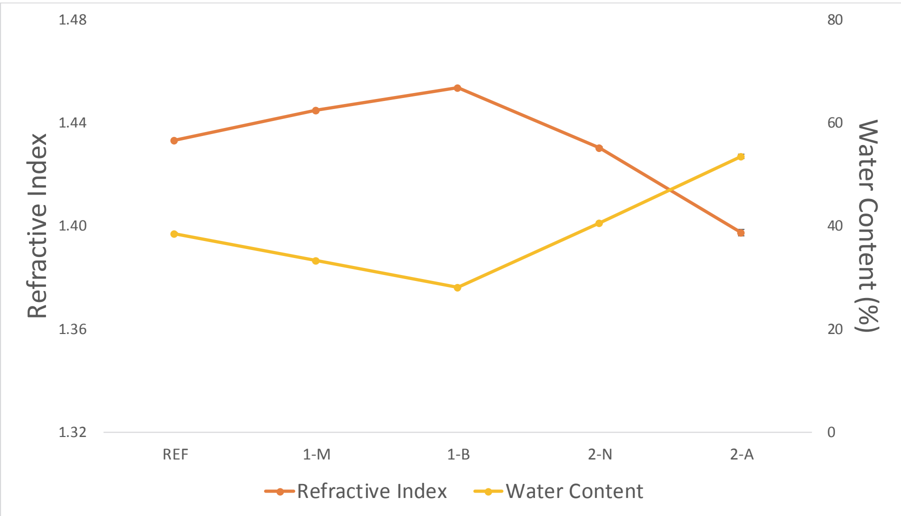
a greater influence on the water content than the addition of NVP.(Ye, K.H., Kim, T.H and Sung, A.Y., 2008) Fig. 3 shows the refractive index and water content according to the additives.

4. Wettability

For the measurement results of the wettability of the fabricated contact lenses, the contact angle of the sample fabricated with the hydrophobic substance slightly increased overall compared to that of REF, and the contact angle of 2-M in the sample fabricated with the hydrophilic substance decreased. The 2-N group, however, showed values similar to those of REF, and the wettability values of 1-M and 1-B, consisting of hydrophobic monomers, were not significantly different. Table 2 shows the contact angles of all the samples.

When the water content and the wettability were compared, the water content was found to have greatly increased or decreased depending on the properties of the added monomers, while the wettability was not significantly changed in all the samples except for the 2-A sample. The impact of the sample is consid-

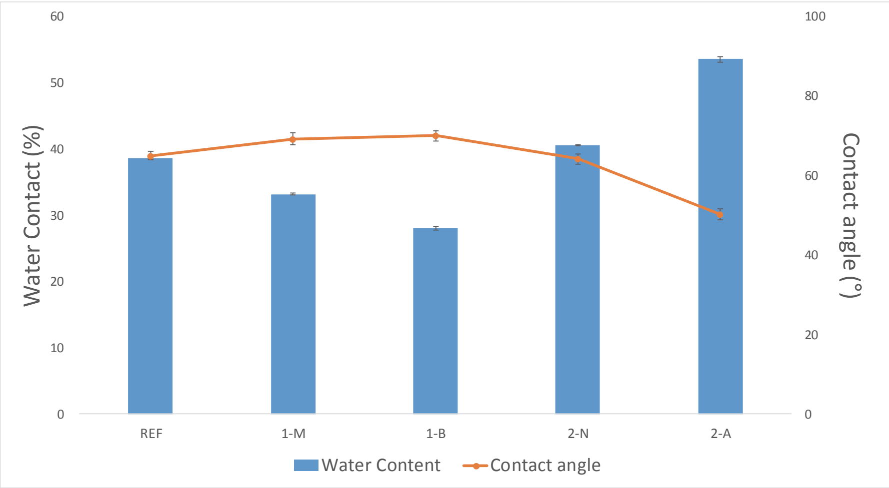
〈Fig. 3〉 Refractive Index and water content of samples



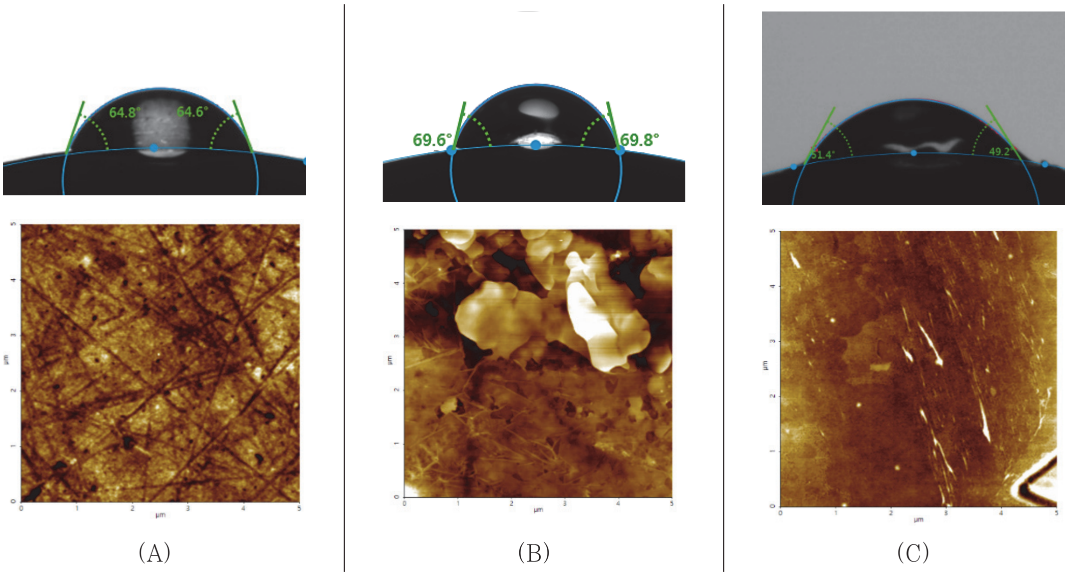
〈Table 2〉 Contact angle of samples. (Unit(°))

Sample	REF	1-M	1-B	2-N	2-A
Contact angle	64.96	69.16	69.94	64.15	50.26

〈Fig. 4〉 Water content and contact angle of samples



〈Fig. 5〉 Contact angle and AFM of samples (A) REF (B) 1-B (C) 2-A



ered insignificant. Fig. 4 shows the water content and contact angle according to the additives.

5. AFM Analysis

To evaluate the surface shape and roughness of the fabricated contact lenses, the REF, 1-B, and 2-A samples were analyzed. For the results of the AFM measurement, the Ra value was 1.715 nm for REF, 3.583 nm for 1-B, and 0.560 nm for 2-A, and the roughness of 1-B with hydrophobicity increased, while that of 2-A with hydrophilicity decreased. As a result, the surface roughness measured using an AFM was found to have affected the wettability, and the AFM and the contact angle seem to have the same tendency, similar to the results of previous studies. (Yang, L., Guo, J., Wua, J., Yang, Y., Zhang, S., Song, J., An, Q. and Gong, Y., 2017; Jang, J.K. and Shin, H.S., 2010) Fig. 5 shows the contact angle and AFM results.

Ⅳ. Conclusion

In this study, to analyze the wettability of hydrogel lenses, hydrogel lenses were fabricated by copolymerizing methyl methacrylate (MMA), butyl methacrylate (BMA), N-vinyl-2-pyrrolidone (NVP), and methacrylic acid (MA) with basic hydrogel contact lens materials, and then their physical properties were evaluated. Overall, the water content decreased in group 1 and increased in group 2 compared with REF containing HEMA, EGDMA and AIBN, further decreased when BMA rather than MMA was added, and further increased when MA rather than NVP was added. The change in the wettability level was insignificant, however, except for the group with MA, so it was considered difficult to judge the wettability level through the water content. The surface roughness measured with an AFM showed the same tendency as the wettability, indicating that the surface roughness affects the wettability. Thus, it is expected to affect the wearing comfort of contact lenses. Therefore, the wettability measured based on the contact angle is an important factor for determining the comfort of the eye when the contact lens is worn. The current standardized specifications for contact lenses, however, do not include the wettability, and it is considered that it needs to be established.

References

Anthony, P. and Lynne, S. (2006). Contact Lenses, 5th Edition, Butterworth-Heinemann Title, 225-227.

Fatt, I. Chaston, J. (1982). Swelling factors of hydrogels and the effect of deswelling (drying) in the eye on power of a soft contact lens, Int.Contact Lens Clin, 9, 146-153.

French, K. (2005). Contact lens material properties. Part 1: wettability, The Optician, 230, 20-28.

Jang, J.K. and Shin, H.S. (2010). Composition and Surface Analyses of RGP Contact Lenses, J. Korean Oph. Opt. Soc., 15(4), 329-337.

Kim, H.J., Kim, S.H. and Kim, J.M. (2011). A Study on Improvement of Wettability and Comfort in Contact lens with Hyaluronic acid, J. Korean Oph. Opt. Soc., 16(3), 255-264.

Kim, T.H., Ye, K.H. and Sung, A.Y. (2008). A study on the measurement of water content hydrogel contact lens by gravimetric method, J. Korean Oph. Opt. Soc., 13(4), 58-63.

Lee, C.W., Jeong, H.W., Kim, H.J. and Ryu, G.C. (2017). Physical Characteristics and Protein Adsorption Properties of Hydrogel Lenses Functionalized with an Interpenetrating Succinyl-Chitosan Network, *Korean Journal of Vision Science*, 19(3), 313-322.

Lorentz, H., Rogers, R. and Jones, L. (2007). The impact of lipid on contact angle wettability, Optom Vis Sci., 84(10), 946-53.

Lee, M.J., Lee, H.M. and Sung, A.Y. (2019). Study on the High Functional Contact Lens Material with Superior Oxygen Transmissibility and Antibacterial Properties, *Journal of Nanoscience and Nanotechnology*, 19(8), 4406-4413.

Lee, M.J., Shin S.M. and Sung, A.Y. (2019). Preparation and Characterization of Functional Ophthalmic Polymer Containing Nanoparticles Using Heat Treatment Method After Polymerization, *Journal of Nanoscience and Nanotechnology*, 19(8), 4495-4502.

Lee, M.J., Sung, A.Y. and Kim, T.H. (2014). Influence of Wetting Agents on Physical Properties of Soft Contact Lens, J Korean Ophthalmic Opt Soc., 19(1), 43-49.

Masnick, K.B., Holden, B.A. (1972). A Study of water content and parametric variations of hydrophilc contact lenses., Aust J Optom., 55(12), 481-487.

Naduvilath, T., Papas, E.B. and Lazon de la Jara, P. (2016). Demographic Factors Affect Ocular Comfort Ratings During Contact Lens Wear, Optom Vis Sci., 93(8), 1004-10.

Read, M.L., Morgan, P.B., Kelly, J.M. and Maldonado-Codina, C. (2011). Dynamic Contact Angle Analysis of Silicone Hydrogel Contact Lenses, J Biomater Appl., 26(1), 85-99.

Tranoudis, I. and Efron, N. (2004). Parameter stability of soft contact lenses made from different materials, Cont Lens Anterior Eye, 27(3), 115-31.

Yang, L., Guo, J., Wua, J., Yang, Y., Zhang, S., Song, J., An, Q. and Gong, Y. (2017). Preparation and properties of a thin membrane based on sodium alginate grafting acrylonitrile, RSC Adv., 7, 50626-50633.

Ye, K.H., Kim, T.H and Sung, A.Y. (2008). Study on Characterization of Contact Lens with Hydrophobic Monomers, J. Korean Oph. Opt. Soc., 13(3), 29-34.

Ye, K.H., Kim, T.H and Sung, A.Y. (2008). The Effects of Hydrophilic Monomers on Water Content of Silicone Hydrogel Contact Lens, Korean J. Vis. Sci. 10(2), 123-130.

논문접수일 : 2019. 05. 27
1차수정본접수일 : 2019. 06. 11
게재확정일 : 2019. 06. 13

표준인증안전학회지(2019. 6) JOURNAL OF STANDARDS, CERTIFICATION AND SAFETY
제9권 제2호 p.55-65.

안의료용 하이드로겔 렌즈의 습윤성 평가에 관한 표준화

신수미* · 성아영**

요 약

본 연구는 콘택트렌즈의 물리적 특성 중 하나인 습윤성의 표준화 규격에 대한 필요성 평가하는 것을 목적으로 하였다. 렌즈의 습윤성에 대한 표준규격의 필요성을 분석하기 위해 기본 하이드로젤 콘택트렌즈 재료에 습윤성 정도가 다른 물질들을 첨가하여 제조된 콘택트렌즈의 물성을 비교하였다. 렌즈제조를 위해 기본적인 모노머인 HEMA (2-hydroxyethyl methacrylate)와 교차결합제인 ethylene glycol dimethacrylate (EGDMA), 개시제인 azobisisobutyronitrile (AIBN), 그리고 친수성 물질로 알려진 N-vinyl-2-pyrrolidone (NVP), methacrylic acid (MA)와 소수성 물질로 알려진 methyl methacrylate (MMA), butyl methacrylate (BMA)를 각각 첨가하여 공중합 하였다. 또한 제조된 콘택트렌즈의 기본 물성을 평가하기 위해 굴절률, 함수율, 접촉각, AFM 및 분광 투과율을 측정하였다. 본 연구의 결과, 함수율의 경우 REF에 비해 MMA와 BMA를 첨가한 하이드로젤렌즈는 각각 약 14%, 27% 감소하였고, NVP와 MA를 첨가한 하이드로젤렌즈는 각각 약 5%, 28% 증가하였다. 습윤성의 경우 REF에 비해 MMA와 BMA를 첨가한 하이드로젤렌즈는 약 6%감소하였고 NVP와 MA를 첨가한 하이드로젤렌즈는 각각 1%, 23%로 증가하는 것으로 나타나 MA를 첨가한 하이드로젤렌즈를 제외하고는 변화가 미미한 것으로 나타났다. 습윤성은 콘택트렌즈 착용감에 영향을 주는 중요한 요소이지만 현재 정확하게 표준화된 규격이 없는 상태이다. 또한 표준화규격에 있는 함수율의 결과와 습윤성의 결과는 비례하지 않는 것으로 나타났으므로, 습윤성을 표준화시켜 정확한 성능 평가를 위해 규격 제정이 필요하다고 판단된다.

※ 주요어: 표준화 규격, 습윤성, 함수율, 콘택트렌즈, 표면거칠기

* 대구가톨릭대학교, 안경광학과, 학생 (soomi8712@naver.com)
** 대구가톨릭대학교, 안경광학과, 교수 (say123sg@hanmail.net), 교신저자