Introduction of UV/EB curable high refractive index monomers and oligomers for optical films

Earl Emerson, Woo geun Kim, and Won Bae

Miwon Specialty Chemical Co., Ltd. 49, Wonsi-ro, Ansan-si, Gyeonggi, Korea

e3@mwc.co.kr / woogeun@mwc.co.kr / baewon@mwc.co.kr

Abstract

The back light unit (BLU) is a core component of liquid crystal displays (LCDs) that are used in notebook computers, mobile phones, navigation devices, flat screen TVs, and public information display (PID) devices, among many others.
To enhance the optical efficiency of LCDs, optical films with a high refractive index have been used in the BLU.
In particular, high refractive materials have been the subject of recent investigations to enhance the optical efficiency of BLUs.
We studied and will present the effect of various monomers and oligomers with high refractive index on optical film prism sheets.

Keyword: UV curing, high refractive index

Introduction

Recent technological trends in thin film transistor-liquid crystal displays(TFT-LCDs) include thinning, lightening, decreasing power consumption, and decreasing costs.
In particular, low power-consuming products are in high demand worldwide because of increased energy-saving awareness.
The electrical power consumption of a TFT-LCD is determined primarily by the back light unit (BLU). To ensure brightness uniformity and high luminance, the BLU is composed of several kinds of optical materials, namely a light source, reflective sheet, light guide plate (LGP), diffuser sheet, prism sheet, and a mold frame as shown schematically in Fig.1 [1–11].
Therefore, high refractive materials play an important role in determining LCD efficiency.
Sulfur and bromine-containing polymers have been developed for advanced integrated optical applications [12–14]. However, due to increasing environmental issues, halogenated materials are forced to replace with halogen-free high refractive materials [15].
Until now, high refractive index materials, which contain Br, have been used mainly as a component resins for prism layer of prism sheet. It is allowed no longer by the restriction of the use of hazardous substances and harmful materials.

For achieving high refractive index materials and following these environmental policies and restrictions at the same time, we developed monomers which contain aromatic group.

To improve the optical efficiency of LCDs, biphenyl acrylates and bisfluorene diacrylate are currently used, though there is great interest in further enhancing the refractive index and mechanical properties of the refractive materials used in the BLU of LCDs.

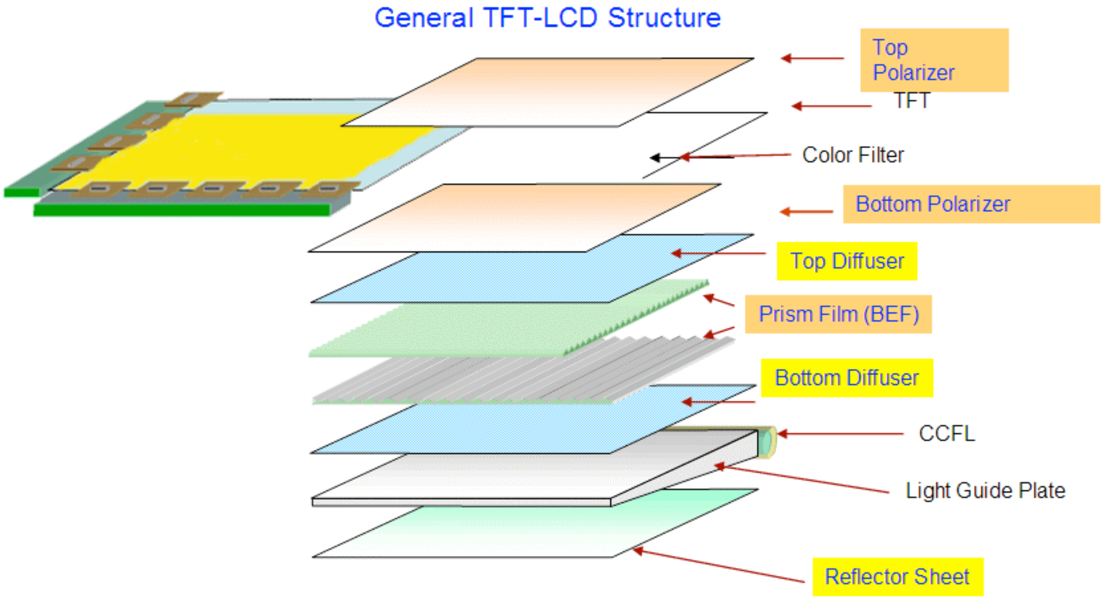


Fig. 1 Structure of a liquid crystal display-back light unit (LCD-BLU)

Background

Flat panel display industry had grown to the extent of $1,15 billion at 2010 year, it presented an average growth rate of 20% per annum from 2002 to 2010. Such growth could be derived from increasing usage volume of FPD, new display device, instead of CRT, existing display device.

To the extent, FPD technology was including creation of new applications and markets (such as thin TV, smart phone and table PC), but not limited to replacement of CRT as existing technology.

Looking at global market situation, the proportion of TVs which adopted FPD as display device was 8.5:10, judging from the global sales amount at 2010.

In detail, in the largest markets such as North America, West Europe, South Korea, the proportion of TVs that didn't adopt FPD merely appeared from 2009.

All this, as well as in China market, the proportion of TVs which adopted FPD as display device was 90% or more, judging from the global sales amount in the fourth quarter of 2010.

In the wave of growth, BLU market, as shown in Fig.2, accomplished high level growth remaking average rate of 10 % from 2007 to 2012 and reached $ 20 billion at

2012 year.

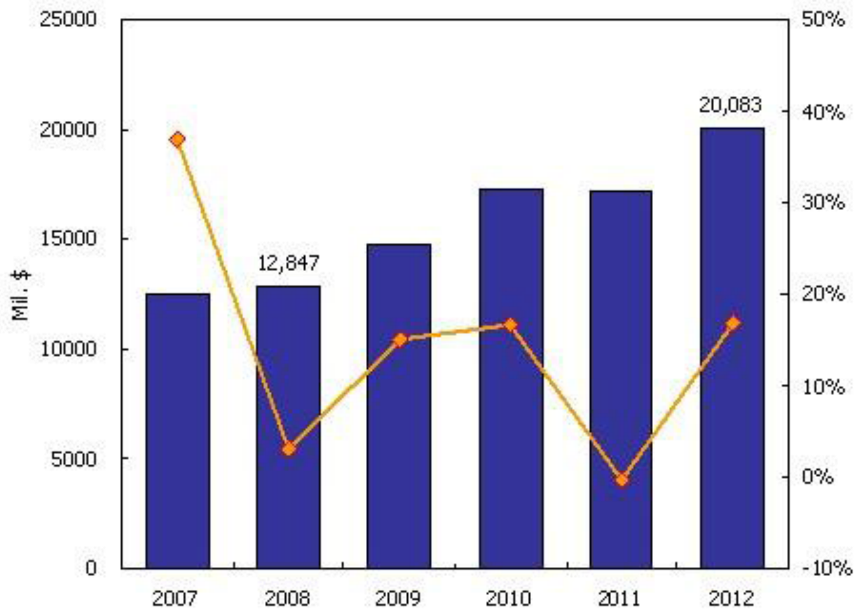
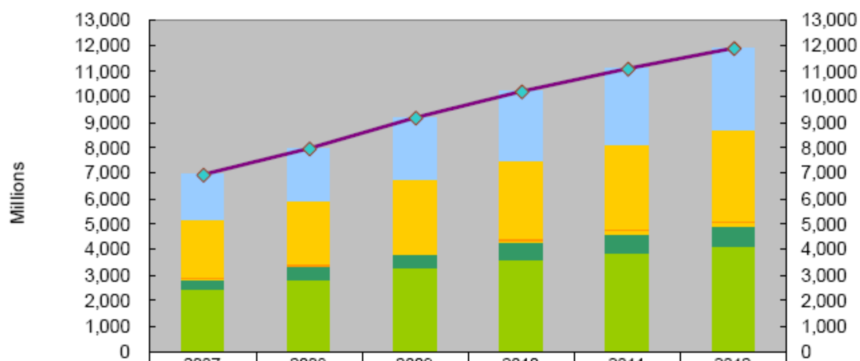


Fig. 2 Market revenue in BLU (Source: Displaybank report)

TFT-LCD panels are built from several components including glass substrates, liquid crystal, plastic frame and optical films. Optical films play an important role in the TFT-LCD module in aiding in the control, diffusion and brightness enhancement of light and in contrast ratios.

Fig.3 is related with BLU from which we can find market information of optical film. In this information, we are focusing on prism sheet which needs high R.I. from which we are developing various materials.



	2007	2008	2009	2010	2011	2012
Reflector Film	1,776.8	2,061.8	2,427.4	2,731.2	2,982.5	3,214.8
Diffuser Film	2,228.4	2,456.5	2,782.2	3,070.4	3,313.8	3,573.4
Reflective Polarizers	72.4	56.2	59.4	56.6	58.0	58.2
Micro Lens Film	44.1	83.6	96.8	113.4	132.7	137.3
MF-Prism	345.2	462.2	565.8	655.6	733.4	797.5
Normal Prism	2,462.1	2,822.4	3,247.2	3,577.2	3,848.4	4,107.9
Total	6,929.1	7,942.7	9,178.7	10,204.5	11,068.9	11,889.0

Source: Displaysearch :Q3`08 Quarterly LCD Components Report

Fig. 3 BLU optic film demand forecast

Prism film is a micro-replicated prism structure film assembled in the LCD backlight module to enhance luminance. BEF (Brightness Enhancement Film) is also called lens film or prism sheet. BEF is 3M's marketing name for prism sheets. The base film of BEF is polyester (PET) or polycarbonate (PC).

This product relies on internal refraction and subsequent re-reflection to direct off-angle light to the front of the display.

A single sheet of prism film can redirect off-angle light toward the viewer coming from one direction only (horizontal or vertical as seen by the display viewer), depending on the alignment of the prisms. The off-angle light from the other direction still comes through an off-angle.

This can be addressed by stacking two prism films one on top of the other: one for the horizontal off-axis light and one for the vertical, as shown below.

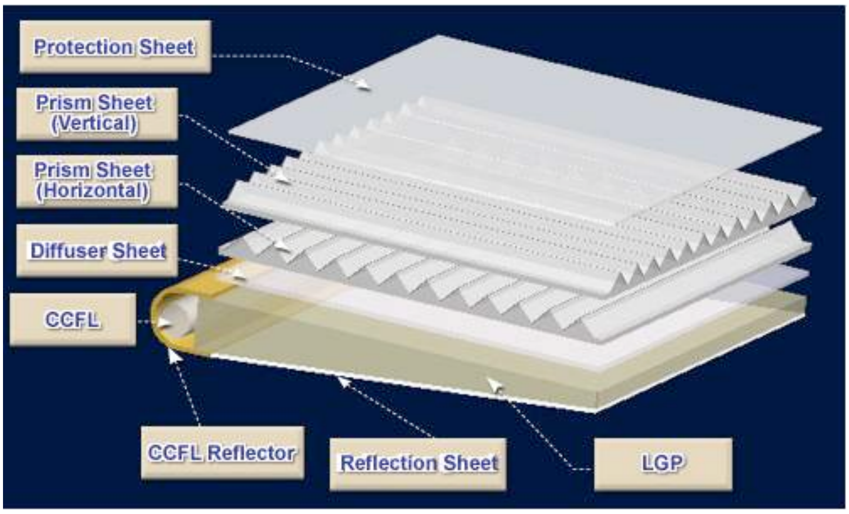


Fig. 4 BLU structure

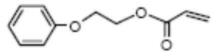
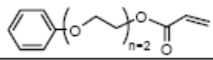
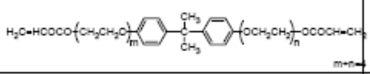
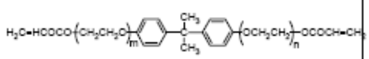
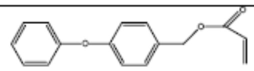
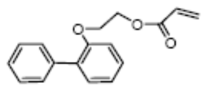
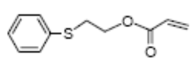
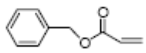
Experimental & Results

Part I. High refractive index monomers & Oligomers

The refractive index depends on the polarizability of the material; therefore, it is desirable to maximize the dipole moment per unit volume induced by the electro-maganetic field.

Substances containing more polarizable groups (e.g., bromine, iodine, sulfur and phosphorus atoms or aromatic rings) or which have a high polarizability over a large atomic area will normally have higher refractive indexes than substances containing less polarizable groups or than strongly electronegative substituents (such as fluorine, oxygen or alkyl groups) will have lower refractive indexes.

Table 1. High refractive index monomers (I)

Product name	Chemical name	Structure	Viscosity (cps, 25℃)	Mw	Specific Gravity	Refractive Index (at 25℃)
M140	1-Ethoxylated phenol acrylate		20	191	1.10	1.516
M142	2-Ethoxylated phenol acrylate		30	226	1.11	1.510
M240	Bisphenol A ethoxylated acrylate		1,000	516	1.14	1.537
M2100	Bisphenol A ethoxylated acrylate		700	773	1.12	1.516
M1122	Phenoxy benzyl acrylate		20	254	1.12	1.565
M1142	1-Ethoxylated-*o*-phenylphenol acrylate		130	268	1.13	1.577
M1162	Phenylthioethyl acrylate		15	208	1.11	1.560
M1182	Benzyl acrylate		5	162	1.06	1.5140

i) Aromatic group

Bulky conjugated or aromatic substituents have a high refractive index. Therefore, a molecule that contains multiple aromatic rings may be expected to have an even higher refractive index. Bisphenol-A epoxy diacrylate has a refractive index of nD25 = 1.557.

While this product has a relatively high refractive index, its viscosity is relatively high, 4,500 cPs @ 60°C. It will be almost impossible to apply such a product by standard coating techniques. For this reason, a low-viscosity monomer with a single-ring structure was developed.

ii) Halogen

Halogenation is a well-known approach to high-refractive index materials. A very typical halogenated molecule having a very high refractive index (nD20) of 1.70 is pentabromophenyl methacrylate. However, this molecule has a poor compatibility/solubility with other molecules because of its very high halogen content. A brominated version of Bisphenol-A epoxy diacrylate has been developed which has a relatively high refractive index, nD25 = 1.588 along with good compatibility with many other UV resins. Recently, there are globally fast-growing demands for only halogen-free materials with high refractive index because of the increasing environmental concerns. Achieving equivalent RI without the use of halogenated materials is a major challenge.

Table 2. High refractive index oligomers

Product name	Chemical name	Appearance	Func.	Viscosity (cps, 60℃)	Tg (℃)	R.I. (25℃)	Type
HR1139	Bromo epoxy acrylate	Liquid	2	1,100	95	1.588	Halogen
HR1162	Bromo epoxy acrylate	Liquid	2	1,350	85	1.581	
HR2200	Epoxy acrylate	Liquid	2	2,000	83	1.559	Non-halogen
HR3000	Urethane acrylate	Liquid	2	1,450	78	1.571	
HR3200	Urethane acrylate	Liquid	4	1,300	97	1.565	
HR3700	Urethane acrylate	Liquid	2	50,000 (25℃)	59	1.585	
HR3800	Urethane acrylate	Liquid	3	1,160	52	1.573	
HR4000	Urethane acrylate	Liquid	2	60,000 (25℃)	61	1.582	

iii) Hetero or sulfur

Many conventional sulfur containing molecules, particularly sulfur-containing aromatic molecules, are characterized by high optical transparency, high dielectric constant, good adhesion to substrates and high refractive index. One well-known example is bis(4-methacryloylthiophenyl) sulfide, or 4,4'-thiodibenzenedithiol dimethacrylate, which has very a high refractive index (nD20) of 1.66. However, it is in a solid state at room temperature and, more important, it has a very limited solubility/compatibility with other UV resins. We have developed proprietary technologies that enable us to synthesize a series of heteroatom containing aromatic urethane acrylate oligomers. These oligomers have a high refractive index, nD25=1.58 (liquid), and excellent optical transparence. Additionally, these oligomers are compatible with many commercial UV resins, thus making them easily formulated.

Table 3. Dilution power of monomers

HR2200

	Contents (%)	Viscosity (cps,25℃)	R. I (25℃)		Contents (%)	Viscosity (cps,25℃)	R. I (25℃)
M140	10	31,830	1.5534	M1182	10	16,700	1.5533
	20	3,940	1.5488		20	2,350	1.5486
	30	944	1.5444		30	310	1.5438
	40	485	1.5408		40	93	1.5396
M240	10	82,280	1.5574	M1162	10	31,070	1.5589
	20	57,100	1.5530		20	3,470	1.5582
	30	31,100	1.5504		30	922	1.5578
	40	16,100	1.5491		40	340	1.5572
PE110	10	61,600	1.5552	M1142	10	62,200	1.5615
	20	33,700	1.5520		20	34,500	1.5633
	30	9,580	1.5472		30	10,800	1.5650
	40	4,500	1.5444		40	4,900	1.5671
M200	10	23,800	1.5480				
	20	3,950	1.5362				
	30	1,055	1.5250				
	40	340	1.5156				

Prism sheet is adopted by high R.I. formulation which requires low viscosity value. Therefore, we are cognizing dilution power as of importance for high R.I. formulation. From Table 3, we can find viscosity value when blending each monomer to HR2200 at 10~40%, in which HR2200 is corresponded to di-functional epoxy acrylate.

The highest quality of dilution power is observed at M1182 and M200. However, in case of M200, it tends to be reduced in R.I formulation, therefore it is barely used for optical film application

Dilution power is observed to be good in M140, M1162, however there is unique odor which makes its application be restricted.

In consideration, the most important point is to which the refractive index can be raised.

Relating this point, a measurement reveals that occasion employing M1142 is raised to the highest level in R.I. formulation.

In market, the most favorite is OPPEA when taking dilution power, refractive index into general account.

Table 4. High refractive index monomers (II)

Product name	Chemical name	Viscosity (cps, 25℃)	R.I. (25℃)	*Mw* (Theoritical)	Dilution (wt%)
HR6042	Bisphenol fluorene diacrylate	20,000	1.600	550	OPPEA 40
HR6060	Modified bisphenol fluorene diacrylate	80,000	1.584	730	-
HR6100	Modified bisphenol fluorene diacrylate	11,000	1.562	900	-

Recently, there is increasing demand for more slim display and more cost down. By which there is increasing efforts in order to reduce number of optical film and enhance brightness.

Among those efforts, there is a trial to remove protecting film which covers outside of optical film and use film as it is.

Then, not to damage prism sheet's mountain, to develop a product having enhanced hardness and scratch resistance is being asked. It is very difficult work to increase Refractive index and keep scratch resistance at the same time.

In the course of that, for satisfying with being asked, we finally reach development of products with enhanced elasticity by means of treating EO addition into fluorene structure whose refractive index is at high level.

The products, which treatment of EO addition is given to fluorene structure, are HR6060, HR6100

One more important factor is hardness of optical film coating. We performed pencil hardness evaluation by means of coating to PET film which is used for prism sheet.

There is difference of primer treatment depending on each PET, by which there is occurrence of difference in surface energy after the coating.

It causes difference of pencil hardness depending on PET maker.

Entirely, the most excellent result is found in HR3200. Its functionality is higher than other oligomers as confirmed from Table 5. This is the reason why such result is drawn.

Table 5. Film properties of high R.I. oligomers – Pencil hardness (I)

	HR1139	HR1162	HR2200	HR3000	HR3200	HR3700	HR3800	HR4000
(1)	2H	2H	2H	3H	4H	3H	3H	2H
(2)	3H	3H	3H	3H	3H	3H	3H	3H
(3)	3H	2H	2H	3H	3H	3H	3H	3H

- Film thickness : 10μm
- Measurement : YOSHIMITSU Pencil tester
- Condition : #400 sand paper, 1kg, 45˚, 30mm/min
- (1) : Toyobo PET (125μm)
- (2) : Kolon PET (188μm)
- (3) : TSI PET (188μm)

Besides, as seen from Table 6, the type where treatment of EO addition is given (such as HR6060, HR6100) is measured to be lower relatively in hardness. But it doesn't mean scratch property is lacking and it plays a role to keep prism sheet's mountain, by courtesy of elasticity improvement.

When comparing HR6100 with HR6060, the former is more treated by EO addition than the latter by which the flexibility of former is more improved.

Table 6. Film properties of high R.I. oligomers – Pencil hardness (II)

	HR6042	HR6060	HR6100
(1)	2H	H	H
(2)	3H	H	B
(3)	3H	H	F
(4)	3H	2H	H
(5)	3H	H	H
(6)	4H	2H	H

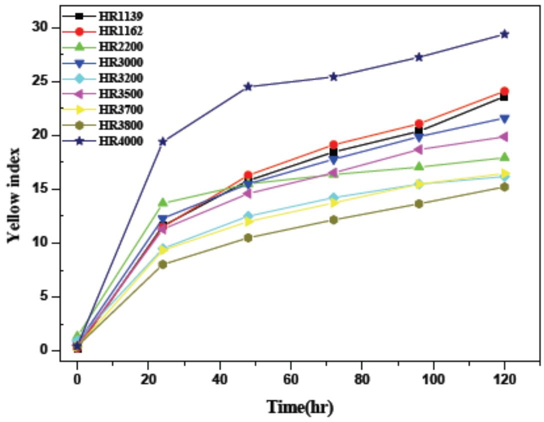
- Film thickness : 10μm
- Measurement : YOSHIMITSU Pencil tester
- Condition : #400 sand paper, 1kg, 45˚, 30mm/min
- (1) : Fuji PET (188μm)
- (2) : TSI PET (188μm)
- (3) : Kolon PET (188μm)
- (4) : Toyobo PET (188μm)
- (5) : SKC PET (SH34M, 188μm)
- (6) : SKC PET (SH34P, 188μm)

Table 7. Film properties of high R.I. oligomers – Yellowing (I)

QUV

	Yellow index(Δ E)					
	0 hr	24 hr	48 hr	72 hr	96 hr	120 hr
HR1139	0.26	11.60	15.80	18.46	20.38	23.60
HR1162	0.26	11.50	16.30	19.12	21.07	24.09
HR2200	1.35	13.70	15.50	16.36	17.05	17.93
HR3000	0.69	12.30	15.50	17.78	19.88	21.61
HR3200	0.99	9.50	12.50	14.22	15.48	16.18
HR3500	0.54	11.30	14.60	16.53	18.68	19.87
HR3700	0.42	9.35	12.00	13.74	15.48	16.46
HR3800	0.46	8.02	10.50	12.17	13.65	15.22
HR4000	0.46	19.40	24.50	25.42	27.24	29.37

- Film thickness : 10 μm
- Measurement : QUV weathering tester – Model : QUV/basic
- Condition : 60℃ & 30 RH



It is found that most of high refractive index material isn't good in yellowing property due to inclusion of a large number of aromatic rings.

For coping with that, trials to improve yellowing property are being done.

In case as to structure where halogen or sulfur compound is contained, yellowing property is worse seriously. What is worse, sulfur is accompanied with odor issue, so the structure is tended to be so far from favorite one.

On the contrary, we make part of aliphatic structure contained in HR3200 and HR3800 by which yellowing property is improved.

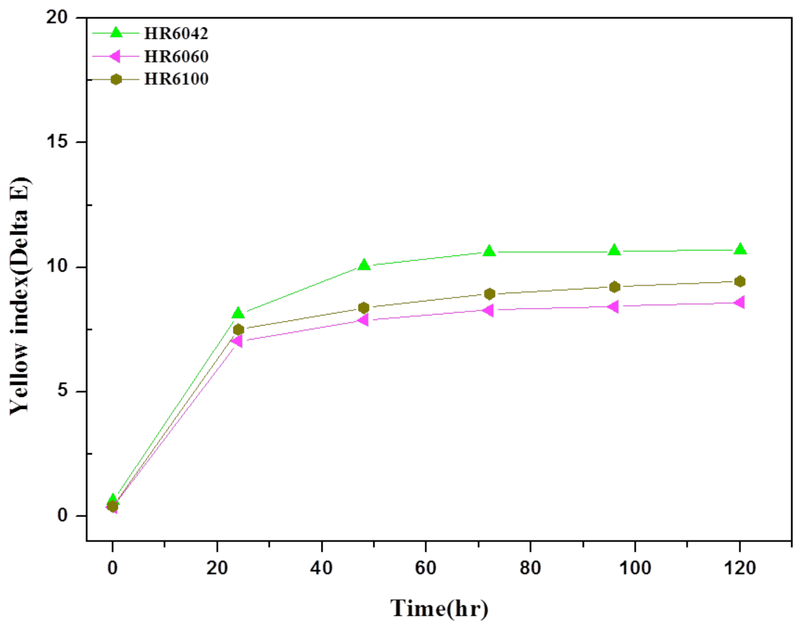
Meanwhile, it is revealed that yellowing property isn't good in case of HR1139, HR1162 due to inclusion of halogen.

Meanwhile, it is revealed that yellowing property isn't good in case of HR4000 due to inclusion of sulfur.

On the one hand, as for HR6060, HR6100, yellowing property is less bad relatively, but number of aromatic ring in the structure is less large relatively by which refractive index tends to be low.

On the other hand, as for HR6042, yellowing property is worse relatively, but it contains OPPEA as a dilution monomer by which refractive index tends to be high.

Table 8. Film properties of high R.I. oligomers – Yellowing (II)



	Yellow index (ΔE)					
	0 hr	**24 hr**	**48 hr**	**72 hr**	**96 hr**	**120 hr**
HR6042	0.63	8.11	10.05	10.61	10.64	10.70
HR6060	0.37	7.03	7.88	8.28	8.44	8.59
HR6100	0.41	7.50	8.38	8.93	9.22	9.43

- Film thickness : 10μm

- Measurement : QUV weathering tester – Model: QUV/basic

- Condition : 45℃ & 30RH

Adhesion property is regarded as the most important one for coating application. Then, we performed cross-cut test by means of disclosure under the following condition: time of 120 hours and temperature of 65℃, 95RH.

Adhesion property of HR3700, HR3800 reports good result relatively.

Table 10 presents adhesion property depending on various PET films.

Adhesion property of HR6100 reports good result, the molecular weight of which is high relatively.

Table 9. Film properties of high R.I. oligomers – Adhesion (I)

	HR1139	HR1162	HR2200	HR3000	HR3200	HR3700	HR3800	HR4000
(1)	0/100	40/100	90/100	80/100	100/100	100/100	100/100	100/100
(2)	0/100	0/100	0/100	80/100	20/100	100/100	100/100	10/100
(3)	10/100	60/100	0/100	0/100	0/100	60/100	60/100	0/100

- Film thickness : 7µm
- Tape : 3M 610
- Oligomer : P.I. = 100 : 5
- Condition : 65℃ & 95RH during 120hr
- (1) : Toyobo PET (125µm)
- (2) : Kolon PET (188µm)
- (3) : TSI PET (188µm)

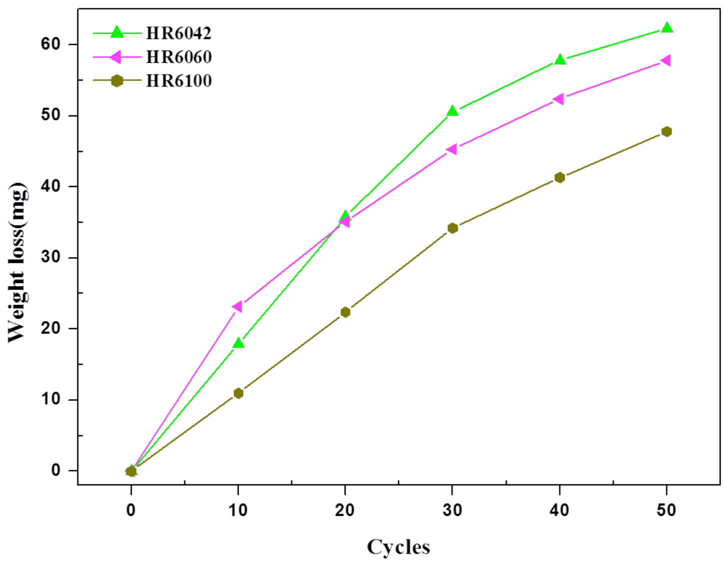
Table 10. Film properties of high R.I. oligomers – Adhesion (II)

	HR6042	HR6060	HR6100
(1)	100/100	17/100	0/100
(2)	0/100	0/100	0/100
(3)	7/100	27/100	88/100
(4)	100/100	0/100	100/100
(5)	100/100	100/100	100/100
(6)	99/100	19/100	99/100

- Film thickness : 7µm
- Tape : 3M 610
- Oligomer : P.I. = 100 : 5
- Condition : 65℃ & 95RH during 120hr
- (1) : Fuji PET (188µm)
- (2) : TSI PET (188µm)
- (3) : Kolon PET (188µm)
- (4) : Toyobo PET (188µm)
- (5) : SKC PET (SH34M, 188µm)
- (6) : SKC PET (SH34P, 188µm)

Through abrasion test, it is confirmed that HR6100, in which treatment of EO addition is done largely for purpose of elasticity improvement, is the most superior item.

However, if excessive treatment of EO addition is done it can be led to refractive index decrease. Therefore the treatment requires proper adjustment to the addition.



- Oligomer : P.I. = 100 : 5
- Film thickness : 40μm
- Measurement : Taber 5150
- Condition : H-18 wheel, 1kg

Fig. 5 Film properties of high R.I. oligomers – Abrasion test

Part II. Organic-Inorganic hybrid materials

A unique, inorganic-organic hybrid nanocomposite technology platform has been developed which significantly improves nano particle compatibility with acrylated resins and, most important, minimizes viscosity increase. Unlike many nano particle dispersions which show various degrees of haziness because of particle agglomerations, this inorganic-organic hybrid nano composite technology demonstrates excellent optical transparency.

Table 11. Organic-inorganic hybrid materials

	SHR1075	SHR1081M2	SHR1111M2	SHR1117M2
Base monomer	M1142	M1122	M1162	M1182HP
Inorganic contents (wt%)	50.0	55.0	55.0	60.0
R.I. (25℃)	1.630	1.650	1.645	1.632
Viscosity (cps, 25℃)	1,000 ~ 4,000	400 ~ 2,000	150 ~500	300 ~ 1,500

Typically, inorganic-organic hybrid nanocomposites can be synthesized by two different methods—(1) solgel process and (2) nanoparticle dispersion in reactive organic medium. In a sol-gel process, semimetal or metal alkoxides, chlorides or nitrates are hydrolyzed and then condensed. The sol-gel process uses a low processing temperature, which allows organic compounds to be homogeneously incorporated into inorganic structures without decomposition.

Unfortunately, neither hydrolysis nor condensation reactions can be completed unless high-temperature processes are used and this often decomposes the organic components or leads to cracking. It has been believed that composites produced by the sol gel method exhibit unstable rheology and coatings formed from them may lack both thermal and hydrolytic stability. Such concerns have prevented use of a large scale of such materials in commercial production. Nano particle dispersion generally comprises inorganic fillers in nano-size, dispersed in organic components such as solvents, UV resins or polymers. However, the dispersions are prone to in-homogeneity, agglomeration and precipitation of inorganic fillers; thus improved stability and life time are highly desired.

A new technology for making nano composites with high refractive index which overcome some or all of the aforementioned problems has been developed. The new nano composite technology involves four features, including nano particle surface modification, nano particle dispersion, nano composite stabilization and formulation of radiation curable, organic-inorganic hybrid nano composite. Nano particle surface modification converts commercially available nano particles that are hydrophilic and non-reactive to nano particles that are hydrophobic and curable. Commercial nano particles are normally aqueous dispersions of inorganic oxides or metal particles. As inorganic nano particles are surface-modified with organic groups, particularly as the surfaces of nano particles are bonded with UV functional groups, the modified nanoparticles become compatible with various radiation-curable monomers and oligomers. Thus, a wide range of radiation-curable, inorganic-organic hybrid nano composites with high refractive index can be prepared.

A unique, inorganic-organic hybrid nano composite technology platform has been developed which significantly improves nano-particle compatibility with acrylated resins and, most important, minimizes viscosity increase. Unlike many nano particle dispersions which show various degrees of haziness because of particle agglomerations, this inorganic-organic hybrid nano composite technology demonstrates excellent optical transparency.

Fig. 6 Organic-Inorganic hybrid materials

Conclusion

Demands are growing faster for new materials which have even higher RI, higher performance and, especially, are halogen-free because of increasing environmental concerns. Achieving equivalent RI without the use of halogenated materials is a big challenge. Based on a broad understanding of the refractive index, new technologies have now been developed which address this challenge, including heteroatom containing materials and inorganic organic, hybrid nano composites. The proprietary surface treatment of the nano materials results in dispersions with the high optical clarity required for optoelectronic applications.

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