Pushing the Limits of LED Curing and Looking Forward to a Bright Future

By Thomas R. Mawby

In recent years, LED curing has become a viable and economical technology for offset printing. In order to maximize the full potential of the LED process, the industry needs to overcome certain technological barriers, such as lamp energy and the lack of suitable photoinitiators. It is now possible to optimize the chemistry and process in order to produce inks for packaging and even low migration inks for primary food packaging.

Background

As the price of oil continues to rise and the necessity for lower environmental impact increases, technology must adapt in order to conform. Energy curable inks and coatings are already known to have one of the lowest environmental impacts, when compared to solvent or waterborne systems, with zero VOCs and minimal energy required to cure. Within the past few years, developments have been made to make UV curing of inks and coatings even more energy efficient, thus reducing the impact on the environment even further.

The commercial use of UV curing inks and coatings has been around for many years and continues to gain popularity based on its many advantages. The biggest of these advantages is the fact that they contain little to no VOCs. By the elimination of the VOCs, UV curing does not need to either recapture or incinerate the exhaust as with traditional solvent/heatset inks and coatings. This also reduces the need for government permits, based on environmental impact. The amount of energy needed to cure a UV ink or coating is much less than that needed to dry solvent/heatset inks and coatings. The instant cure of the UV inks and coatings results in faster turnarounds and secondary finishing directly off press. Although the impact of UV curing on the environment is minimal, there is always room for improvement.

Some of the drawbacks with traditional UV lamps are:

- the lamps contain a small amount of mercury. If they are broken, special care is needed to ensure that the mercury is contained and disposed of safely.
- Because UV lamps emit wavelengths in the UV region below 280 nm, ozone is produced. This requires an outside exhaust to remove the ozone from the production floor.
- In order for the lamps to remain lit, they must stay above a minimum light intensity. If the energy is reduced lower than this, it will result in a lamp that is not producing light.
- It takes time for the UV lamps to come up to full power and, if shut off, they must cool prior to re-firing.
- A high amount of heat is generated which can distort non-porous

substrates, resulting in registration issues. Additionally, it is possible to dry out paper/board substrates causing curl or cracking. To combat heat, the use of dichroic filters (used to remove IR energy), water cooling and air cooling have been utilized.

- The water cooling and air cooling needed for UV lamps, along with the exhaust needed to remove ozone, makes them bulky.
- Efficiency of UV lamps is typically below 15% of the energy used. One of the first technologies to reduce the drawbacks of UV is LED curing.

LED technology has now been introduced into industrial applications. One of the earliest commercial uses of high powered UV-LED was for dental work where curing with UV light of dental adhesives and fillers reduced the time the patient was held in the dental chair waiting for their work to be completed. The small compact size of a LED lamp was able to pinpoint the area needing attention.[1] Another early application was the use of UV-LED for curing adhesives on printed circuit boards. Once again the time to cure the UV adhesives was minimal compared to traditional adhesive curing methods. Additionally, the LED was able to spot cure the adhesive with minimal heat thereby reducing possible damage to the fragile electronics. It was only a matter of time until the UV-LED caught up with the graphic arts market to bring its advantages to the production floor.

UV-LEDs have been around since the 1990's. In 1998, the first "high power" UV-LED was developed and had output wattage of 5 mW/cm^2.[2] It was not until 2006 that UV-LEDs became viable for use in UV curing with lamps that had output wattages of 2 W/cm^2. With increased packing of LEDs, (Figure 1) and cooling technologies that allow the LEDs to run at a reduced temperature (approximately 60° C), the output wattage has increased to 16 W/cm^2.[3]

The efficiency of a medium pressure mercury lamp is approximately <10-15%. Most of the energy produced is converted to IR energy, in the form of heat. With LED lamps, 25-30% efficiency of the energy is converted to useful UV light and the remaining energy consists of heat generation.[4] It is imperative that cooling be used in LED (either water or air cooled). If the LEDs get too hot, it will reduce the lifetime of the LED lamp. As the wavelength of the LED is decreased, for example 395 nm to 365 nm, the efficiency will also be reduced by 25-30%. The energy required to run an LED lamp is 80 Watts/lamp whereas the traditional UV lamp uses 1.2 kW/lamp. This difference in energy use can result in a significant savings.[5]

UV-LED lamps come in a variety of sizes, power and wavelengths. The output spectra of the LEDs are monochromatic. Their outputs only span, at the most, 40 nm with the peak at 365, 385 or 395 nm. (Other UV-LEDs have been produced at 350, 405, 210, 250, 275 or 290 nm). Most of the special output ranges are for specialty applications such as water purification. As a rule of thumb, as the output spectra is decreased so is the maximum intensity of the lamp.[6] A 365 nm lamp at this time has a maximum output of 2 W/cm^2 whereas the 395 nm lamp has a maximum output of 10-16 W/cm^2.

LED lamps also have other factors which make them more desirable than a traditional UV lamps:

- Because the wavelength of the light produced by the LED is not in the area of 280 nm or less, this means that no ozone is produced by these lamps and thus, there is no need for an external exhaust. Traditional UV lamps require exhausting of the ozone.
- LED lamps run at cooler temperatures than traditional UV lamps. A LED lamp will at most produce 60° C of heat whereas mercury lamps will produce heat in excess of 350° C. In order to remove heat, either large amounts of chilled air or water are required.
- The LED lamps are instant on and off. Normal UV curing lamps require time to achieve working output intensity. If the lamps are turned off, then the lamps need to cool prior to re-firing. In order to get around this, lamp manufactures have developed the use of a shutter to help reduce the time between starts and stops.
- Because of the exhaust, cooling and shutters, traditional UV lamps are bulky. LED lamps do not require the extraction of ozone. They do require water or air cooling, but due to the lower temperatures, the size is minimal. There is no need for shutters because the LED is instant on and off. Therefore the size of the LED lamps is much smaller than a mercury UV lamp.
- The LED lamps have been found to have much greater lifespan than normal UV arc lamps. The LED lamps have a working lifetime of >20,000 hours whereas the UV arc lamp is rated normally between 1,000 and 2,000 hours.
- LED lamps contain no mercury where a traditional UV lamp has a small amount of mercury which would have to be disposed of properly.
- Maintenance with LED lamps is also minimal when compared to traditional UV curing lamps. A traditional UV lamp needs to be changed every 700-1,000 hours and the reflectors need cleaning when the bulbs are replaced.

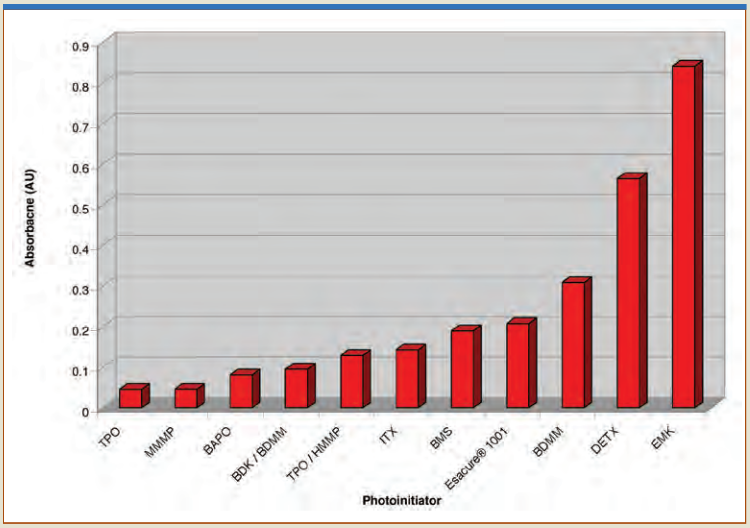
There are some drawbacks with LED lamp technology:

- Due to the limited amount of photoinitiators available, the LED curing of varnishes and coatings becomes more challenging. The photoinitiators that are used to cure these products have a tendency to shift yellow when cured.

- The choice of raw materials (especially the photoinitiators) that can be used for LED is limited and therefore the price of the inks is greater than traditional UV curable inks.
- Since the LED lamps do not contain short wavelength light energy (under 320 nm), most of the curing associated with the lamp will be through cure.
- The LED lamps, at this time, cost almost 3.5 x the amount as a traditional UV lamp. A 110 cm LED lamp would be approximately $102,000 where as the arc lamp would be $28,500.[7] As with any new technology, as the number of units sold increases, the cost will decrease.
- The power output of LED lamps is continually improving, although is still lacking when compared to traditional UV lamps.
- The effects of oxygen inhibition must be overcome to get acceptable surface cure of inks and coatings. Since surface curing is minimal with LED (365 nm and above), the effect of oxygen inhibition plays a greater part in cure. Additional steps must be taken over what is typical in conventional UV curing.

Figure 1

365 nm absorbance of photoinitiators (100 ppm concentration of PI)



In order to develop inks and coating that will cure with LED lamps, the photoinitiator must utilize the energy of the lamp between the 385 to 410 nm. At this time, there are very few photoinitiators which absorb within these wavelengths. This impacts the cost and efficiency of the photoinitiator package. Even though the photoinitiator absorbs in this region, the primary absorption peaks of the compounds are usually less than 385 nm and therefore the efficiency of the photoinitiator to absorb all of the available light at 385 nm is diminished.

As with any UV curable ink or coating, it is necessary to know the output spectra of the lamps and the dosage delivered to the substrate at press speeds, in order to match the photoinitiator package with the lamp's output. Once the lamp information is obtained, it is necessary to find the photoinitiators that absorb at the peak intensity wavelengths of the lamp. By taking the output spectra of each of the photoinitiators, a graph can be made to pinpoint which photoinitiators are viable candidates. For example: Figure 1 shows the photoinitiators that absorb at 365 nm. It can be seen that at 365 nm, EMK (Ethyl Michler's Ketone) gives the greatest absorbance. In order to have an ink or coating that will cure with a 365 nm UV-LED lamp, it would be beneficial to use EMK in combination with other photoinitiators that have appreciable absorbance in the 365 nm output. Additionally, the same technique can be used for other wavelength lamps, such as 385 nm and 405 nm. (Figure 2 and Figure 3) *Please note:* the spectral data of the photoinitiators was obtained at a 100 ppm concentration with spectroscopy grade methanol using a Hewlett Packard Model 8453 UV/VIS Diode Array Spectrophotometer and a 1 cm quartz cell.

When using Norrish type II photoinitiators such as DETX (CAS# 82799-44-8), ITX (CAS#7508121-9), EMK (CAS# 90-93-7), BMS (CAS# 83846-85-9), 4-MBP (CAS# 134-84-9), Esacure® 1001M (CAS# 272460-97-6) and MBF (CAS# 15206-55-0), an amine synergist must be used for hydrogen abstraction to produce a very active donor molecule which carries out the photo polymerization reaction. The reactivity of the type of amines increases in the following way:[8]

$$(C_2H_5)_3N < CH_3N(C_2H_4OH)_2 < (CH_3)_2NCH_2CH(OH)CH_3 < (CH_3)_2NC_6H_4COOC_2H_5$$

Since the effective LED lamp output is toward the high end of the UV electromagnetic spectrum (365 nm,

Figure 2

385 nm absorbance of photoinitiators (100 ppm concentration of PI)

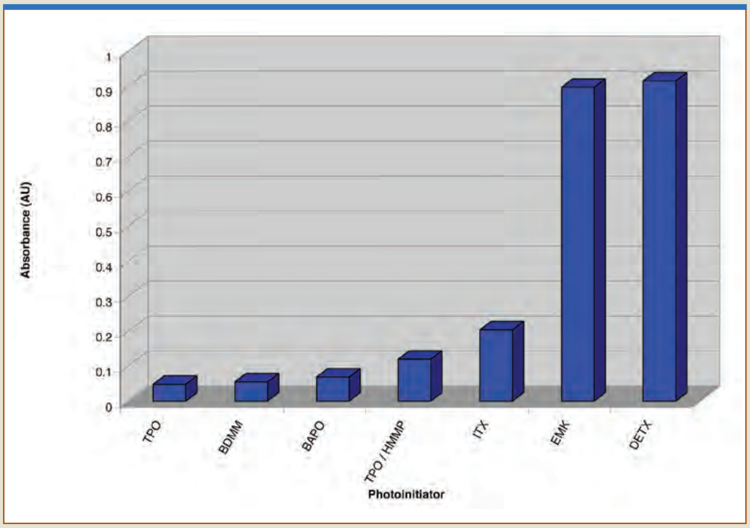
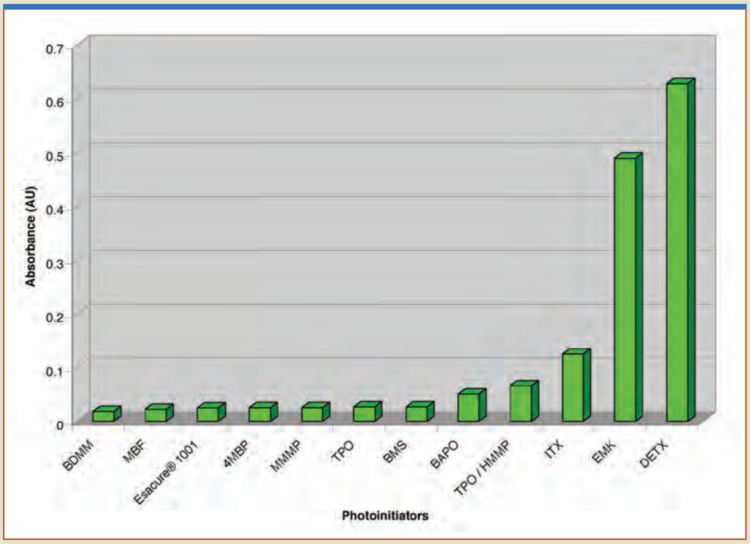


Figure 3

405 nm absorbance of photoinitiators (100 ppm concentration of PI)



385 nm, 395 nm or 405 nm), the amount of surface cure is minimal when compared to through cure. In order to achieve a non-tacky surface, the need to maximize the amount of surface cure, needs to be examined. This would require formulations to minimize oxygen inhibition at the surface of the ink or coating. The most effective ways to offset oxygen inhibition is the following:[9]

- The use of a gas to remove the oxygen at the surface such as nitrogen or carbon dioxide.
- The use of waxes in the ink or coating that provides a surface barrier.
- The use hydrogen donors to quench the peroxy radicals (amines, thiols, ethers, silanes, phosphites).[10]
- A combination of high levels of photoinitiators and high intensity light sources providing an excess of produced photoinitiator free radicals.
- Short wavelength UV light.

If the inks and coating are to be used on food packaging, one must follow applicable regulations for the geographical location. For example, all European regulations must be met for inks and coating which may come in contact with foodstuffs (primary food packaging not intended for direct food contact). This would include all European Council directives and suitable use listings. (i.e., Regulation (EC) No 1935/2004, Swiss Ordinance 817.023.21 Annex 6, Directive 2007/42/EC, Directive 2002/72/EC, Regulation (EC) No 2023/2006, etc.) Along with these regulations, certain end use customers have their own guidance rules for materials used for food packaging inks and coatings. (i.e., Nestle Guidance Note on Packaging Inks[11]) Looking at the suitable photoinitiators in the figures and these listings, mixture of BDK/BDMM (CAS# 24650-42-8/119313-12-1), BMS (CAS# 83846-859), MBF (CAS# 15206-55-0), DETX (CAS# 82799-44-8), ITX (CAS#75081-21-9), TPO (CAS# 7598060-8), MMMP (CAS# 71868-10-5), 4-MBP (CAS# 134-84-9) and mixture of HMMP/TPO (CAS# 75980-60-8 / 7473-98-5) cannot be used for products printed for Nestle. This leaves only BAPO (CAS# 162881-26-7), BDMM (CAS# 119313-12-1), Esacure® 1001M (CAS# 272460-97-6) and EMK (CAS# 90-93-7). In addition to these photoinitiators, certain polymeric photoinitiators can be utilized.

Experimental

The UV-LED curing unit that was utilized for all LED testing was the Air Motion System XP5 and categorized as a 12 W/cm^2 lamp. The output of the lamp was measured with an EIT Power Puck II equipped with UVA, UVB, UVA2 and UVV filters. The output of the lamp running at 100% power, 7.5 cm from the conveyor belt at a speed of 300 fpm, was measured as 2093.9 mW/cm^2 – UVA, 3383.6 mW/cm^2 – UVA2 and 3078.5 mW/cm^2 – UVV with the smoothing setting on the instrument in the "off" position. The delivered dose of the lamp running at these conditions gave a dose of 26.7 mJ/cm^2 – UVA, 43.6 mJ/cm^2 – UVA2 and 40.4 mJ/cm^2 – UVV.

All inks were printed using a Prufbau Printability Tester equipped with UV rollers at an application speed of 0.5 m/s and a pressure of 700 N. The substrate that was used for printing was a standard 14 point SBS board (*Solid Bleached Sulfate*). The inks were printed to the following densities: Black – 1.80, Cyan – 1.40, Magenta – 1.50 and Yellow – 1.05. (The densities of the prints were read with an X-rite 528 spectrodensitometer with Illumination/Observer setting of D50/2°).

The cure of the printed samples was evaluated by using a combination of subjective tests, which are typically performed at the end of a press, and near-infrared spectroscopy. The subjective testing included:

- **Thumb twist**—immediately after curing, the print is subjected to a thumb twist (delivered with great downward pressure) and the amount of ink movement is noted.
- **Scratch resistance**—The print is scratched with a fingernail and the damage to the printed surface is recorded.
- **Cross-hatch adhesion** was performed on all printed samples to assess the cure of the inks using 3M 610 type tape.
- **Cotton ball test**—a cotton ball is rubbed across the surface of the print to check for tackiness of the surface. The amount of fuzz left by the test is quantified. The near infrared spectroscopy is utilized to look at the C-H vibration due to the stretching of the acrylate double

TABLE 1

Lab testing with 12 W/cm^2 395 nm lamp at 100%, 7.5 cm from curing surface and a speed of 300 fpm

	Thumb Twist	Scratch Resistance	Adhesion	Cotton Ball Test	NIR Conversion
Mixture A -Black	3	4	5	3	62%
Mixture B -Black	5	4	5	4	68%
Mixture C -Black	5	4	5	4	72%
Mixture D -Black	4	4	5	4	70%
Mixture A -Cyan	4	4	5	4	65%
Mixture B -Cyan	5	5	5	5	72%
Mixture C -Cyan	5	5	5	5	76%
Mixture D -Cyan	5	5	5	5	74%
Mixture A -Magenta	4	4	5	4	64%
Mixture B -Magenta	5	5	5	5	72%
Mixture C -Magenta	5	5	5	5	74%
Mixture D -Magenta	5	5	5	5	75%
Mixture A -Yellow	4	4	5	4	67%
Mixture B -Yellow	5	5	5	5	76%
Mixture C -Yellow	5	5	5	5	76%
Mixture D -Yellow	5	5	5	5	74%

5 = Excellent 1 = Poor

TABLE 2

Commercial press testing with one 9 W/cm² and one 12 W/cm² 395 nm lamp at 100%, 7.5 cm from curing surface and a speed of 15k impressions per hour

	Thumb Twist	Scratch Resistance	Adhesion	Cotton Ball Test	NIR Conversion
Mixture A -Black	3	4	5	3	53%
Mixture B -Black	5	4	5	4	57%
Mixture C -Black	5	4	5	4	55%
Mixture D -Black	5	4	5	4	55%
Mixture A -Cyan	4	4	5	4	57%
Mixture B -Cyan	5	4	5	5	60%
Mixture C -Cyan	5	4	5	5	60%
Mixture D -Cyan	5	4	5	5	60%
Mixture A -Magenta	4	4	5	4	55%
Mixture B -Magenta	5	4	5	5	60%
Mixture C -Magenta	5	4	5	5	60%
Mixture D -Magenta	5	4	5	5	60%
Mixture A -Yellow	4	4	5	4	59%
Mixture B -Yellow	5	4	5	5	63%
Mixture C -Yellow	5	4	5	5	63%
Mixture D -Yellow	5	4	5	5	63%

5 = Excellent   1 = Poor

bond at 1620 nm. It is the decay of this bond that gives insight as to the extent of conversion of the UV curable materials.[13]

Results

Through a screening design of experiment and subsequent optimize design, the above mentioned photoinitiator compounds and amine synergist were tested. A total of four different combinations were identified as having proper cure when utilizing the UV-LED 395 nm lamp at the 7.5 cm distance. Each mixture was tested in all four process colors in a UV curable offset formulation. See Table 1 for results.

These mixtures were then press tested on a commercial UV-LED offset press equipped with two 395 nm UV-LED lamps at output wattages of 9 W/cm² and 12 W/cm². Testing was conducted at both 10k impressions per hour and 15k impressions per hour. The first test was run with both lamps at 100% power and 15k impressions per hour. The results can be found in Table 2. The second test was performed using the two lamps at 100% power and 10k impressions per hour. The results can be seen in Table 3.

From these mixtures, it was determined that the inks developed for offset UV-LED printing are a viable option and could be used for packaging applications. A variation of the photoinitiator was then made, utilizing the European Council directives and suitable use listings mentioned above, to see if a primary food packaging UV-LED offset ink could be made and pass the migration/extraction testing.

The inks were made in the laboratory and tested according to *EN 14338: Paper and Board intended to come into contact with foodstuffs. Conditions for determination of migration from paper and board using modifiedpolyphenylene oxide (MPPO) as a simulant. and EuPIA Guideline on Printing Inks applied on the non-food contact surface of food packaging materials and articles—September 2009.* The testing was conducted utilizing both GC/MS and LC/MS to ensure that the lowest possible quantifiable limit of 10 ppb was met for all ingredients in the formulation. *Please note:* In

TABLE 3

Commercial press testing with one 9 W/cm^2 and one 12 W/cm^2 395 nm lamp at 100%, 7.5 cm from curing surface and a speed of 10k impressions per hour

	Thumb Twist	Scratch Resistance	Adhesion	Cotton Ball Test	NIR Conversion
Mixture A -Black	4	4	5	4	55%
Mixture B -Black	5	5	5	5	62%
Mixture C -Black	5	5	5	5	60%
Mixture D -Black	5	5	5	5	60%
Mixture A -Cyan	5	4	5	4	57%
Mixture B -Cyan	5	5	5	5	65%
Mixture C -Cyan	5	5	5	5	65%
Mixture D -Cyan	5	5	5	5	62%
Mixture A -Magenta	5	5	5	5	62%
Mixture B -Magenta	5	5	5	5	65%
Mixture C -Magenta	5	5	5	5	67%
Mixture D -Magenta	5	5	5	5	67%
Mixture A -Yellow	5	5	5	5	64%
Mixture B -Yellow	5	5	5	5	67%
Mixture C -Yellow	5	5	5	5	67%
Mixture D -Yellow	5	5	5	5	65%

5 = Excellent 1 = Poor

order to ensure that the cure of the inks are at optimum level, each ink must be cured prior to the next ink being applied. Knowing this and the traditional press sequence of colors, the black ink was cured four times, the cyan ink was cured three times, the magenta ink was cured two times and the yellow ink was cured one time through the lamp at 100% power and 300 fpm. This would simulate the amount of lamps each ink would see on press. See Table 4 for the results of the migration/extraction testing.

Conclusion

It is now possible to produce offset printing inks that can utilize the advancements in UV-LED lamp technology. Not only is it possible to develop inks that can be used for packaging applications, it is also possible to provide inks that can be used as a primary food package (for certain food types). As the technology continues to grow, the ability to utilize these curing processes will become more commonplace. The lamp manufactures are making great strides by doubling the output lamp energies approximately every two years. The use of differing wavelengths will also be key. At this point, the only lamps that can deliver the output necessary are based on 395 nm lamp technologies. Work is continuing on lamps whose output spectrum is 365 nm, 300 nm, 285 nm and 265 nm. At this time, the power and reliability are not quite there but recent advancements are making these LEDs a possibility in the near future. In addition to the advancements in the lamp technologies, advancements will have to be made in the photoinitiators that absorb in the UV-LED wavelengths. Not only will they have to produce free radicals, but they must also provide pathways for low migration/extraction inks and coatings. With all of the benefits of this technology, along with the advancements to overcome the disadvantages, (i.e., lamp output, available photoinitiators, etc.) the future of this technology is now, and like the lamps that are utilized, only getting brighter.

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TABLE 4

Quantified extractables from print—indirect contact, Tenax GR 60/80 as food simulate (units = µg of substance/kg of foodstuff)

Sample Board	Irradiated Board	Black	Cyan	Magenta	Yellow	LQL(ppb)
PI 1	<LQL	<LQL	<LQL	<LQL	<LQL	10
PI 2	<LQL	<LQL	<LQL	<LQL	<LQL	10
PI 3	<LQL	<LQL	<LQL	<LQL	<LQL	10
PI 4	<LQL	<LQL	<LQL	<LQL	<LQL	10
Monomer 1	<LQL	<LQL	<LQL	<LQL	<LQL	10
Monomer 2	<LQL	<LQL	<LQL	<LQL	<LQL	10
Oligomer 1	<LQL	<LQL	<LQL	<LQL	<LQL	10
Oligomer 2	<LQL	<LQL	<LQL	<LQL	<LQL	10
Additive 1	<LQL	<LQL	<LQL	<LQL	<LQL	10
Additive 2	<LQL	<LQL	<LQL	<LQL	<LQL	10

µg/kg food corresponds to µg/6 dm2, assuming that 1 kg of food is in contact with 6 dm2 of packaging

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