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User's Manual and Technical Information

What is NuPlon[™] Resin?

NuPlon[™] resin is a proprietary curable pre-polymer comprised of hydroxyl and acid units which, upon heating, react with one another forming a chemically crosslinked solid by releasing a water molecule. The reaction is reversible as adding water back to the reaction mixture breaks the bonds and chemically degrades the structure down into small, non-toxic components. Typical degradation time is 2-3 months of exposure to water under moderately warm temperatures (86 °F, or 30 °C).



Figure 1. Schematic chemical reaction of formation and degradation of NuPlon[™].

Pre-polymer Properties

The prepolymer is a viscous, clear, off-white liquid which can be poured out and handled as a liquid. It is medium viscosity at room temperature ($\sim 1500 - 2500$ cP, Brookfield model LVDVE viscometer, #31 spindle, 5 RPM 20 °C)



Figure 2. NuPlon[™] prepolymer resin liquid.

The liquid can be poured into any open-top mold. As the liquid is adhesive and can form tight bonds, Akina has had best success with flexible silicone molds coated with PTFE mold release spray (VDX, Microcare or equivalent <u>https://medical.microcare.com/products/vdx-dry-lubricant-spray/</u>). Additionally, thin, disposable metal molds may also be used. It is important for the molding assembly to have access to air to allow for release of water reaction by product to occur during curing.

<u>Safety</u>

Prior to curing, the prepolymer is an oligomeric mixture of short polyesters along with carboxylic acids and multi-functional alcohols. One of the major components is lactic acid, which contributes to the slightly 'vinegar-like' smell of the prepolymer. As lactic acid is moderately caustic, it is advised to avoid contact of the NuPlonTM prepolymer resin with skin or eyes as well as it should not be ingested.

For curing processes, the substance must be heated. Follow all safety instructions provided by the manufacturer of the heating equipment you use for this process. As it cures, NuPlonTM will off-gas a small amount of lactic acid, which may be irritating to the eyes and nose. For this reason, curing should be performed only in well ventilated areas.

Curing

The process of curing NuPlonTM requires removal of water. This can be accomplished in an oven set to 130 °C (266 °F) or shorter times at higher temperatures up to ~ 170 °C (338 °F) above which darkening can occur. the typical curing time applied at Akina has typically been overnight (~10 – 24 hrs) for temperatures ~150 - 170C or over 2-3 days for temperatures ~120 – 150 °C. An indication of successful crosslinking is the formation of a solid which remains solid even at the curing temperatures (i.e. is not a liquid at 130 °C). As part of the curing process, it is important for the material to have good access to air-flow to evaporate away water. Note that whatever mold this curing occurs in will be the final shape of the material and it is suggested for the mold to be level to the ground to prevent pooling/puddling of the liquid.



Figure 3. Curing process of forming NuPlonTM circle with "NuPlonTM" etched in the bottom of the mold.

The NuPlon[™] can be optionally treated under vacuum to degass and vacuum can be used to assist in water removal. Despite this, the temperature must still be high enough for the chemical condensation reaction to occur and vacuum does not change this. As an example, treating the NuPlon[™] precursor under vacuum (-31 inHg) at 90 °C overnight yielded a strongly adhesive viscoelastic material which was only partially crosslinked. As a side note, the application of purposefully performing partial-crosslinking the polymer to generate biodegradable

adhesives remains a viable option for applications which need an adhesive. After the material is removed from the oven it can be cooled and demolded.



Figure 4. NuPlon[™] cured in a silicon pan (partial fill, cured in shape that liquid was in after being added.).

As water is removed from the reaction of curing the typical finished cure mass is less than the initial masses with common yield around 70% (weight final piece/mass added resin).

Alternate Curing

In the absence of a laboratory oven, NuPlonTM can also be cured using conventional household equipment such as crock-pots or other heaters. Testing at Akina, Inc. has determined that the NuPlonTM can be cured in a typical crockpot (Intertek Model # SCR450-5) with the crockpot set to 'low' setting on the dial (internal chamber temperature measures as ~ 123 °C) overnight.



Figure 5. Curing NuPlon[™] in a conventional store-bought Crockpot.

Processing

Unlike thermoplastic polymers, NuPlon[™] will not melt even when exposed to heating. This makes most conventional machining techniques quite effective with no jamming, gumming or other problems common to machining of plastic.





Figure 6. Example of machining NuPlon[™] into fork using conventional bandsaw/sander.



Figure 7. Example of cutting NuPlon[™] into a hair-comb.

This is only a few examples of machining of NuPlon[™] and other shaping techniques can be used to obtain a wide-variety of products.

Properties

Optical



Figure 8. NuPlon[™] piece resting on top of grid-paper.

NuPlonTM presents good spectral transparency within the visible light region. The data below is for type M317 NuPlonTM based on % transmittance through a 2 mm thick section of plastic.

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NuPlon™	n TM % Transmittance at wavelength (nm)								Thickness
Type	200	300	400	500	600	700	800	900	(mm)
M317	0.1	0.8	72.9	87.1	88.6	88.9	89.0	87.8	2.0

Table 1. Transmittance (%) of light at indicated wavelength.



Figure 9. NuPlonTM UV-Vis Transmittance graph (type M317).

Given that transmittance drops to near-zero below 300 nm, NuPlonTM can be used to apply for ultra-violet shielding and protection.

Mechanical

Cured NuPlon[™] type M317 in dry state is a hard, firm substance with the following mechanical properties

Property	Value
Elastic Modulus $(0.1 - 1\%$ strain)	4.8 <u>+</u> 1.6 MPa
Tensile Strength	31.0 <u>+</u> 19.2 MPa
Extensibility	5.3 ± 1.6 % strain

Table 2. Mechanical properties of cured NuPlonTM (dog-bone test pieces, Tensile tested on TA.XTplus (Texture Technologies) at a crosshead speed of 1 mm/sec.) presented as average \pm standard deviation, N = 2.

Thermal

NuPlon[™] has unique properties in regards to its thermal behavior. Unlike thermoplastic materials, NuPlon[™] does not melt nor does it become especially brittle with cold. Based on testing at Akina, NuPlon[™] has been determined to be mechanically stable at temperatures between - 78 °C to 350 °C. This broad range of temperature stability applies only to the piece as it is dry due to its ability to be permeated by and affected by water.



Figure 10. Touching hot soldering iron to piece of cured NuPlon[™] (type M317).

The thermal conductivity of NuPlonTM is relatively poor which enables it to be used as an insulator. It's insulative properties can be enhanced by incorporation of sodium chloride (salt) at roughly 10 - 30% w/w of precursor before curing. Curing with sodium chloride creates a highly porous matrix which traps air and improves NuPlonTM's thermal resistance.



Figure 11. Salt-cured NuPlon[™], holding piece against soldering iron.

Degradability/Disposal

Within 1 day of exposure to water, NuPlon[™] will become a highly plasticized, flexible material. It will remain whole, but flexible for up to 2-3 months (depending on temperature/water condition) after which point it will start to split up and break down eventually hydrolyzing to non-toxic water-soluble compounds. Testing at

Akina, Inc. has indicated that type M317 NuPlonTM loses $93 \pm 1\%$ (Average \pm STDEV, N = 3) of its mass after 2 months incubation at 30 °C with orbital agitation at 100 RPM in locally collected pond water.

Additionally, NuPlonTM, although not especially flammable, can be burned without formation of toxic fumes outside of typical carbon dioxide and other smoke components. Exposure to boiling hot water quickly forms a multitude of cracks within the NuPlonTM making it easy to break apart into smaller portions which degrade more quickly and are amenable to disposal.



Figure 11. Examples of wet and dry NuPlonTM, respectively.



Figure 12. From left to right: NuPlon[™] piece original in water, after 2 months, remaining solids on piece of filter paper after passing NuPlon[™] degraded residue through.

Customizability

Incorporation of varying additives can be used to modify the NuPlonTM materials properties to provide for a wide array of properties. Contact John Garner (jg@akinainc.com) to discuss customization opportunities to meet your needs.

Licensing Considerations

The NuPlon[™] platform technology is PATENT PENDING with a priority date of filing established from provisional filing on June 25, 2020. The user's guide presented here contains information from laboratory testing at Akina, Inc. In no way does it represent all the potential uses and considerations for the NuPlon[™] material. Contact John Garner (jg@akinainc.com) for joint development and licensing opportunities.