

# Rubber-to-Substrate Bonding Agents Technical Guide

## General Use of Specialty Adhesives – Water-borne Bonding Agents



## Introduction

MEGUM™ & THIXON™ bonding agents have been used for more than 80 years to successfully bond rubber-to-metal. Today, these adhesives are used in bonding a variety of elastomer compounds to metal and plastic substrates, including steel, stainless steel, aluminum, zinc, copper, polyamides, polyacetals, polyesters, and PTFE (with some of these substrates potentially needing specific pretreatment). MEGUM™ & THIXON™ react in a co-vulcanization process to establish a strong bond between elastomers and substrates. Establishing a strong bond with THIXON™ and MEGUM™ bonding agents requires heat and thus occurs during the vulcanization process.

In this guide, we put the focus on the application of water-borne bonding agents in the manufacturing of rubber-metal-parts, focusing on the most commonly used products. Starting with basic differences between water-borne and solvent-based systems, all parameters and topics relevant for the coating process will be covered in this guide.

### Typically bonded components

MEGUM™ & THIXON™ bonding agents are used in many bonding applications combining the unique properties of rubber and metal (or non-metal) substrate. The resulting rubber-metal parts are used in a wide variety of industrial applications including:

- Vibration control elements: springs, bumpers, bushings, suspension systems, clutch mounts, flexible couplings, and engine mounts
- Corded-rubber belts and hoses
- Seals and gaskets
- Bearing elements for buildings and bridges
- Rollers
- Solid rubber tires
- Track pads for caterpillar vehicles, linings and protective coverings
- Metal-supported profiles

## General description

Solvent-based MEGUM™ & THIXON™ bonding agents are low-viscosity and rely on the dissolution of specific polymers (halogenated polymers for instance) and other reactive chemicals. They are used as one-coat bonding agents or two-coat primer and cover-cement systems. Some products within the range are clear non-pigmented solutions.

However, in the water-borne MEGUM™ W-series, this polymer chemistry is used in form of dispersions. In general, water-borne bonding agents, especially with pigmented dispersions, are a combination of pigments and polymer emulsions in water. These dispersions behave differently compared to solvent-based materials. For this reason, the handling and application of water-borne pigmented systems will be discussed in this guide in more detail.

A summary section, specifically for the use of DuPont water-borne bonding agents can be found at the end of this guide.

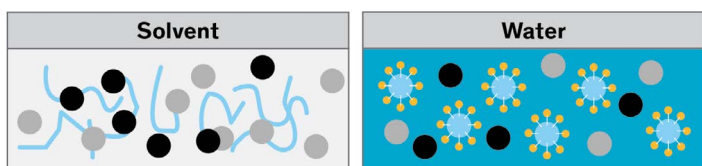
### Basic differences of water-borne and solvent-based bonding agents

In general, all regular solvent-based bonding agents are suspensions – meaning dissolved polymers and some additives in solvents together with insoluble solid substances (pigments). For bonding agents with pigments, both water-borne and solvent-based materials are rather similar in handling and application. However, for water-borne bonding agents, there are some significant differences that will be discussed in more detail in the following sections.

The differences between solvent-based and water-borne bonding agents are mainly related to the difference of polymer occurrence in the product. While polymers can be readily dissolved in solvent-based products, which enables the polymer chains to extend throughout the solution, in most water-borne products, polymers are added as emulsions, to achieve compatibility with the aqueous environment. This procedure adds complexity to bonding agent preparation.

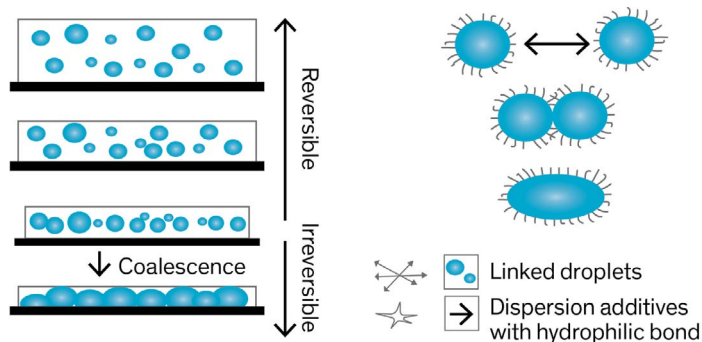
Emulsions are encapsulations of polymer chains in micelles (small droplets), which leads to a stabilization in aqueous dispersions. Micelles are organic phases, which are stabilized by surfactants. Surfactants typically consist of a hydrophilic part (capable to mix with or dissolve in water) creating compatibility with water and a hydrophobic (water repellent) part, that is linking to the polymeric phase. The overall term for mixtures of different phases that are not soluble in each other is called dispersion. A typical example of an unpigmented water-borne dispersion would be milk – a dispersion of oil (fat) in water. Furthermore, mixtures of liquid in liquid (like milk) are called emulsion; mixtures of solids in liquid (like sand in water) are named suspension; and mixtures of liquid in gas (hairspray, for instance) are called aerosol. An overall fact is, that all phases (liquid, solid, gas) are not soluble in each other and exist side-by-side, well dispersed in the complete mixture.

As visualized schematically in [Picture 1](#), it becomes clear that, in solvent-based adhesives, the functional polymeric ingredients form a solution. Polymer solubility however, comes with drawbacks in bonding agent preparation. For example, certain combinations of polymers and additives/cross-linkers cannot be used in solvent-based formulations because they would simply react immediately. In a water-borne bonding agent though, all relevant ingredients are separated from each other as the polymers are concentrated in the micelle and thus separated from other reagents by surfactants. Only upon destruction of the emulsion, the reaction between ingredients may start.



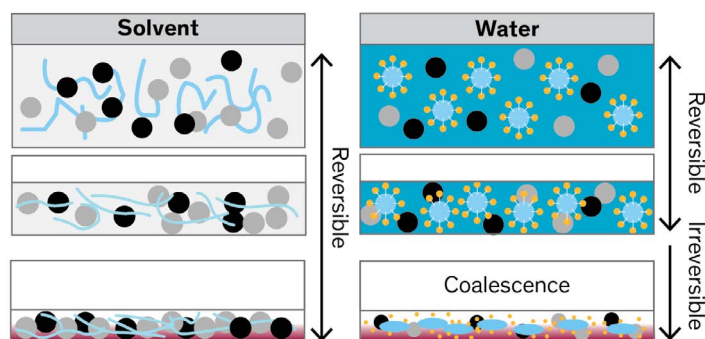
[Picture 1](#): Primary difference of solvent-based and water-borne dispersion.

The film formation, in comparison, is significantly different for that reason as well. Schematically, this formation process is described below in [Picture 2](#). During the drying process, the distance between the individual micelles starts to decrease, and, at a certain point, the droplets are so close that they begin to touch each other which leads to combining the micelles to larger ones (fusion process). Further drying will drive this fusion further until the “point of no return” (coalescence) is reached.



[Picture 2](#): Schematics of drying process in water-borne emulsions.

The comparison of film formation clearly shows the major differences between these two technologies regarding the drying process. In solvent-based bonding agents (despite the pigments), all ingredients are dissolved and can be re-dissolved after the drying. In contrast, water-borne bonding agents, once coalesced, can neither be re-dissolved nor re-dispersed due to the destruction of the emulsion, as seen in [Picture 3](#).



[Picture 3](#): Primary difference in film formation as a summary visualized and compared.

As a result, water-borne bonding agents have a significantly different film formation and film build compared to solvent-based materials. This process of coalescence will happen very rapidly, and, once finished, water-borne films show excellent film integrity achieved through entanglement of the polymeric chains upon breaking of the micelle structure.

## Viscosity

Viscosity, in general, is described as the inner friction and, in case of solvent-based bonding agents, is mainly a function of dry solids (not differentiating the effect of surface, surface chemistry, and shape of the solids), polymer type, and molecular weight of the polymer used, as polymer chains are fully dissolved and thus extended/elongated. The viscosity significantly reflects the influence of dry solids if compared to water-borne systems. In water-borne dispersions, viscosity measurement is significantly less feasible to determine the correct dilution in comparison to solvent-based systems. Here, all ingredients are distinguished and separated, especially the polymers which are enclosed in micelles. These micelles represent the main influence on viscosity. Thus, micelles of the same size will affect in the same way.

In [Table 1](#), a generic overview of water vs. solvent is given.

Table 1: Generic Parameters of Solvent-based vs. Water-borne Bonding Agents

Solvent-based	Water-borne
Polymer solution	Polymer emulsion
Density < 1	Density > 1
Av. DS: 24-28%	Av. DS: significantly higher
Generally applied in dilution	Dilution possible
Ketones/Aromatics	Deionized/Demineralized water
Needs adjustments for electrostatic application	Suitable for electrostatic application
Some 'cleaning' capabilities	Needs clean surfaces (~>45 dyne/cm) to wet

Looking into the homogenization process (e.g., stirring, shaking, tumbling), water-borne products require more delicacy compared to solvent-based materials, mainly to prevent foam build-up. Foam, regarding viscosity, has a significant impact and will falsify the measurement. As already mentioned, water-borne bonding agents require the use of surfactants and, in some cases, wetting agents. The risk of creating "micro" foam is rather significant and problematic. This micro foam requires a very long time to dissipate and will affect not only viscosity, but film formation and appearance as well.

Guidance for how to minimize these potential issues will be provided in the paragraph titled "Storage and Handling Prior to Use."

## Application

### Surface energy and surface tension

In general, MEGUM™ & THIXON™ bonding agents are applied as coatings onto substrates. The latter include mild and stainless steels, non-ferrous metals, plastics, natural and synthetic fabrics and fibers, glass fiber, and ceramics. Typically, a substrate's surface needs a pretreatment to ensure that it is clean and provides a suitable bonding surface for the bonding agent. Plastic materials always require specific pretreatment for the application of water-borne bonding agents.

This pretreatment refers to the timeframe between the cleaning of parts and application of a bonding agent. Here, water-borne materials show higher sensitivity to contamination such as fingerprints, which represent an oily contamination. Unfortunately, water is NOT capable of removing an oily contamination which can significantly worsen the wetting property of a water-borne adhesive. Therefore, keeping cleaned parts contaminant-free is paramount when using water-borne products. Due to their solvent content, solvent-based products do have certain "cleaning" capabilities during application.

Because of the physical and chemical uniqueness of water, substrates require precise cleaning to ensure a high surface energy (~> 45 dyne/cm at the minimum) and a smooth coating process. [The SI unit N/m for surface energy refers to J/m<sup>2</sup> or 1000 dyne/cm, which is the most common unit used to describe surface energy.]

Table 2: Evaporation Numbers and Surface Tension of Typical Liquids

Solvent	Evaporation rate (diethylether = 1) <sup>1)</sup>	Surface tension [dyne/cm] <sup>2)</sup>
MEK	2.6	~25
MIBK	7	~24
(o,m,p)-Xylene	17	~28-32
Toluene	6.1	~44
Ethanol	8.3	~21
Iso-propanol	11	~22
MPA	34	~28 <sup>3)</sup>
Water	~80	68-73 <sup>4)</sup>

<sup>1)</sup> [Lehrbuch der Lacktechnologie, 4. Aufl., Farbe und Lack Ed.; Kap.: 2.2 – Vincenz Verl 2012]

<sup>2)</sup> [Umweltbundesamt Stoffdatenbank: www.stoffdaten-stars.de]

<sup>3)</sup> [BASF Techn. Merkblatt; M 5888 d; Okt. 2010; on-line]

<sup>4)</sup> for water this refers to be a function of temperature

In Table 2, not only the evaporation number, but also the surface tension value exposes drawbacks of using water as a solvent. First, drying a water-borne adhesive requires more energy, as the evaporation number is significantly higher than for commonly used solvents. In addition, the cleanliness of a high-energy surface or the conversion of a low-energy surface to a higher energy is required to assure the surface tension of water is overcome and the spreading/wetting of the surface with water is possible. Second, the listed surface energies – here “surface tensions” for liquids – indicate the significant difference of water compared to most commonly used organic solvents.

As a liquid will only wet surfaces with higher surface energies than their own surface tension, it is obvious that a focus on cleaning for high-energy surfaces, like metal or glass, is required to allow a good wetting of water. On the other hand, if a low-energy surface is to be wetted, the surface tension needs to be increased, in order to enable wetting with water (see Table 3). A measurement, based on this fact, is the methodology to determine the contact angle from liquids on solid surfaces. This method shows very clearly how the interaction of the different molecules of the liquid and solid surface influences the behavior of liquids regarding spreading across the wetted surface.

Table 3: Critical Surface Energies of Solids

Material	Surface energy <sup>1)</sup> [dyne/cm]
PTFE	~18
pp <sup>2)</sup>	~29
PE-LD	~31
PA 6	~43
PA66 <sup>2)</sup>	~47
Al	~1200
Fe	~2500

www.Kern.de/de/Kunststofflexikon/Oberflaechenenergie (online); 'Kritische Oberflächenenergie von Kunststoffen und Vergleichsproben'

<sup>1)</sup> (for information only and depending on liquid for measurement)

<sup>2)</sup> Polymer Interface and Adhesion; Souheng Wu; 1982; Chapter 3

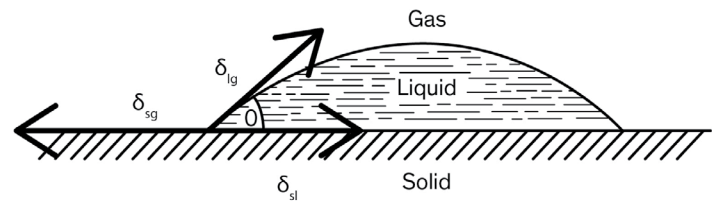
Mathematically, this phenomenon of contact angle and the different interactions between the molecules of each area has been described by the Young's equation (Picture 4):

$\theta$  = Contact angle

$\delta_{lg}$  = surface tension of the used liquid

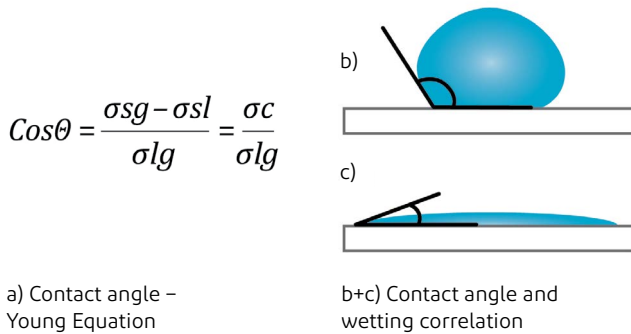
$\delta_{sg}$  = surface energy of the solid

$\delta_{sl}$  = energy of interphase C = critical surface energy



Picture 4: Model for the assessment of wettability (G. Wedler, Lehrbuch der physikalischen Chemie, 1985, VCH Weinheim, 379-380)

To understand this more vividly, [Figure 1a](#) shows the contact angle measurement to determine either the surface tension of an unknown liquid if the surface energy is known, or the surface energy of a solid surface in case the surface energy of the liquid is known. In addition, it clearly describes the forces between the different materials. If the interactive forces between the liquid molecules are higher than the interaction between liquid molecules and surface atoms, the liquid molecules tend to be more attracted to themselves, creating a large angle toward the surface ([Figure 1b](#)). On the other hand, if the attraction between liquid molecules and surface atoms is higher, the liquid molecules are 'pulled' towards the surface, the liquid film spreads, and the liquid "wets" the surface, thus creating a small contact angle ([Figure 1c](#)).



**Figure 1:** Young equation and contact angle wetting correlation.

As described in [Table 3](#), low-energy surfaces, like polyethylene or in the extreme PTFE, require a treatment to adjust to higher levels for wetting with water-borne bonding agents. Here, plasma or corona treatment are widely used methods. Even nylon, by itself, needs to be treated in order to ensure wettability with water. Luckily, most nylon compounds used in this industry contain fillers, like glass fibers, which increase the surface energy to a level that enables wetting of water, if cleaned thoroughly to assure a careful removal of processing aids or release agents used in the manufacturing process of the nylon part itself. Sometimes, when surface energies are borderline (as with nylon), roughening the surface can create enough interaction capabilities to support wetting with water-borne bonding agents. However, MEGUM™ W-series bonding agents contain wetting agents which help lower surface tension.

## Substrate cleaning

Referring to the previous chapter, it is obvious that for water-borne bonding agents, the cleanliness of the substrate surface is critical for the success of the application. Please refer to the Substrate Preparation **Water-borne Bonding-agents Guide** for more details. For now, simply refer to the "Water-break-free" test which allows an easy check on a potential existing contamination issue or insufficient cleaning process.

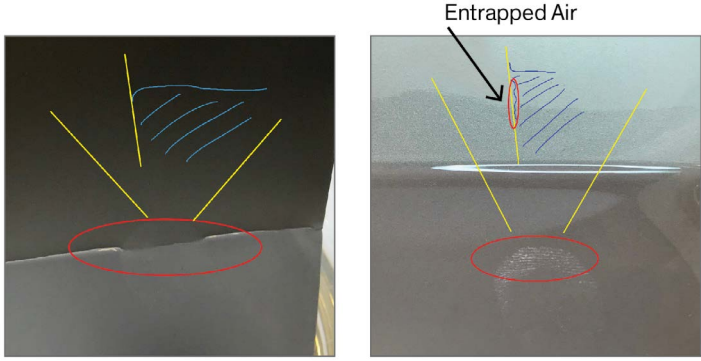
This test mainly makes use of the behavior explained in the above section. Water tends to spread and wet surfaces when they are clean and have sufficient levels of surface energy. [Picture 5](#), showcases the dramatic difference in the wettability between a contaminated and a clean metal surface. Insufficient cleaning can immediately be detected by the wettability pattern.



**Picture 5:** Water-break-free test. Insufficiently cleaned surface (left), well-cleaned surface with water film (right).

To test this in a different way, a surface can be checked by slowly dipping the part in question into a container with clear water and monitoring the liquid meniscus. If the meniscus creeps up the surface or just shows little wrinkles along the line, the surface is ready for the first and possibly latter stages for water wetting. In case the meniscus is drawn into the water while dipping the part further down, it is a clear sign for insufficient surface energy to be wetted with water ([Picture 6a](#)). When the part is completely immersed, the non-wetted area will show up as a shiny little silver pearl ([Picture 6b](#)) thus indicating an area of entrapped air where the water was not able to replace the air on the surface. (Please refer also to our [Substrate Preparation Guide – Water](#))





Picture 6a: Water meniscus seems to be drawn into the water

Picture 6b: Entrapped air inside the water due to lack of wetting

### Drying after coating

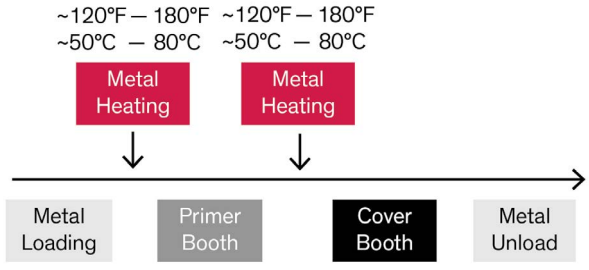
Due to the fact that evaporation happens at the interface to the air environment, the drying of emulsions versus solutions will become critical for this application within a short time period.

During a regular drying step, the concentration of water will decrease faster from the outside to the bulk of the adhesive layer. Hence, the concentration of the emulsion decreases quickly from the outside toward the bulk of the material, accelerating coagulation from the outside-in. This, however, may create a potential skin at the border to the air environment, which can then entrap the remainder of water inside the adhesive layer.

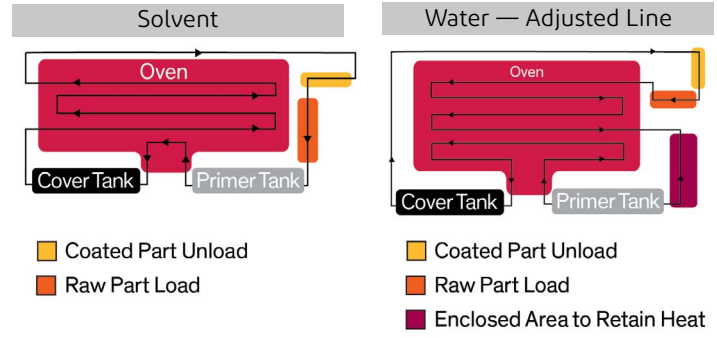
During vulcanization, this water will begin to boil, creating very typical shaped areas of metal or rubber/cover failure. These areas look like blisters in the bonded area of the part when destroyed.

To overcome this behavior, the process needs to be adjusted to make sure water is driven out of the adhesive layer, for instance, by preheating the substrate before the actual coating process. This will not only reduce the risk of over-skinning, but also, of creating rust on the coated metal, in case of primer coating as well.

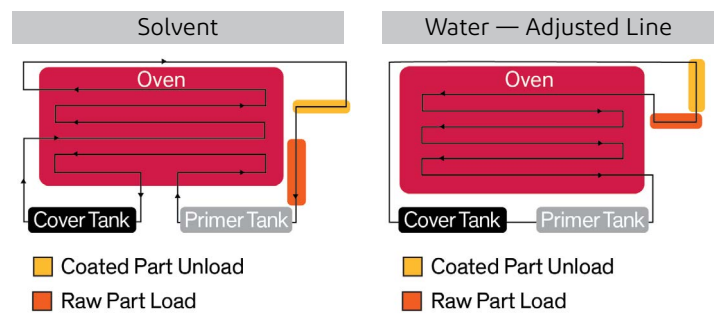
In general, we recommend applying water-borne bonding agents onto substrates which have been heated up to 50°C – 80°C / 120°F – 180°F. See Picture 7 for a generic scheme of the application process and Picture 8a–e for some ideas how to use existing line set-up and how to convert from a line for solvent-based bonding agent application to a line suitable for water-borne bonding agents, with only minor adjustments. The option depends on the size and type of the part in focus as the heat capacity of the overall part determines the potential set-up.



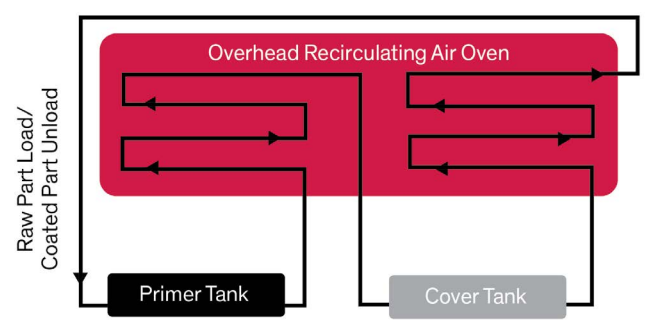
Picture 7: Generic process for application of water-borne bonding agents



Picture 8a+b: Examples of line adjustments to convert to water – small parts – chain-on-edge



Picture 8c+d: Examples of line adjustments to convert to water - large parts - chain-on-edge



Picture 8e: Examples of line adjustments to convert to water dipping line

Although most application processes can be adjusted coming from a regular chain-on-edge application for solvent-based adhesives, some points need to be considered:

- A) Water-borne bonding agents are NOT TOLERANT of insufficient metal cleaning.
- B) Depending on part geometry and size/mass, the preheating process must be adjusted differently reflecting the part's thermal conductivity (the ability to heat up and keep the temperature over time).
- C) As dispersions are shear sensitive, not all delivery systems, suitable for solvent-based materials, can be used for water-borne products:
  - => for delivery pumps, a double-membrane pump is highly recommended
  - => in-line back-pressure regulators must not be activated through circulation of the material
  - => high shear conditions in a circulation line must be prevented (right-angle plumbing)
- D) As water-borne systems will coagulate and cannot be re-dissolved, the delivery piping needs to be kept in continuous flow and/or equipped with a possibility to be flushed with DI-water regularly (Use of spray guns designed for water-borne coatings is optional).
- E) Spray guns need to be flushed when stopping the line, to prevent coagulation of material in spray nozzles.
- F) In case of dipping lines, as water does not dissolve organic contaminants, they will float and coat immersing parts each time; hence, unused tanks need to be covered and protected from organic contamination.
- G) Water-borne application plays by a different set of rules; hence, production staff need to unlearn habits acquired from working with solvent-based adhesives.

## Line preparation for water-borne bonding agents

To prepare an existing application line for the use of water-borne bonding agents, these steps need to be followed precisely:

- 1) Check all materials in contact with bonding agent regarding water suitability to **avoid corrosion**
- 2) All materials in contact with water need to be either **plastic or SST**
- 3) Check delivery equipment for **suitability to handle dispersions**
- 4) **Clean bonding-agent line** thoroughly with solvent used to dilute the bonding agents
- 5) **Flush line with MEK**
- 6) Make sure MEK is not allowed to stay in the line for more than a couple of minutes
- 7) **Flush line with DI-water** until the rinse is clean

Flushing with water is essential, as any remainder of solvent will cause coagulation (coalescence) and plug the line!

In case the line will be used for solvent-based materials again, follow the steps in reverse. Replacing the lines for use of each material (solvent/water) is recommended and absolutely preferred to minimize cross contamination.



## Application – Generics

### Hybrid systems

In general, hybrid systems – the combination of water-borne and solvent-based bonding agents – are possible.

Either a solvent-based primer together with water-borne cover or vice-versa can be applied if enough drying of each coating is secured. For example:

- A) Using a water-borne primer requires the preheating of the part to secure the inside-out drying for this coating. Lower temperatures may be utilized, but, depending on the drying state, might not require additional heating for the drying of the solvent-based cover cement after it has been applied.
- B) Using a solvent-based primer requires thorough drying/ preheating of the part after the primer has been applied to assure the required inside-out drying of the water-borne cover after it has been applied.

Please refer to your technical contact for further guidance and support.

## Application – Primer Specifics

### Thixotropy

MEGUM™ W 9300 and MEGUM™ W 9301 are water-borne primers with excellent environmental resistance. Both are unique in their behavior as well.

Thixotropy describes a phenomenon where viscosity is a function of shear – generally speaking, a function of stirring speed. Specifically, with increasing shear/stirring speed, viscosity decreases. These two water-borne primers (depending on storage history) can appear, in the first run, as a pudding and may seem to be gelled. In this case, this appearance and behavior is **wanted**, as it stabilizes the material during storage and reduces settling.

To reach the consistency required for successful application, the material needs to be stirred using a stirrer (Picture 9 in “stirring” section) with high initial speed until a vortex is formed. During this process, the whole pudding will slowly start moving and the consistency slowly begins to change to the state desired of low viscosity. At this point, the stirrer speed needs to be reduced **immediately** to prevent foaming.

This entire process of breaking the thixotropy takes just a couple of minutes, and then the material can be stirred as any other adhesive without further attention. Adding a little water (~1-2%) will speed up the degradation of the pudding to a normally-behaving liquid. If the material has been initially stirred and the thixotropy has been broken, the primer will stay in this consistency for several weeks or even months depending on age and dilution.

**Caution:** These primers will require consistent stirring during application (as any other bonding agent whether solvent- or water-borne), as partial settling will happen even while in low viscosity!

## Storage and Handling Prior to Use

### Storage

MEGUM™ W-series bonding-agent containers should be stored in dry conditions, indoors and protected from direct sunlight. It is essential that water-borne products are prevented from freezing, as emulsions will be destroyed when frozen!

DO NOT USE MATERIAL THAT WAS PREVIOUSLY FROZEN.

### Avoid evaporation

After use, containers should be completely closed to minimize water evaporation and to prevent the ingress of contaminants. Evaporation of water will occur from open-topped MEGUM™ W bonding-agent containers, but significantly slower than with solvent-based products where gradually the product viscosity will increase. Although evaporation in water-borne agents will not cause an immediate reduction in product effectiveness, with increasing dry solids, the potential for coagulation will become more serious. Coagulation will destroy the product and prevent it from further use.

If necessary, MEGUM™ W-series products can be diluted to a specific preferable density/dry solids content (as viscosity is less suitable for dilution ratio determination/ monitoring). The use of de-ionized or de-mineralized water as diluent is essential. **Tap water is not recommended** due to its mineral content.

Please contact your technical specialist for further details.

### Agitate before use

Most MEGUM™ W-series bonding agents contain dispersed solids that can settle during storage. It is important to displace and re-disperse any solids settlement by adequate and frequent stirring. Bonding-agent containers should be stirred using an electric or air-driven motor fitted with a propeller.

For water-borne bonding agents, some specifics regarding re-homogenization need to be followed:

Using a paint shaker should NOT be considered! Foaming will be accelerated and the possibility of creating micro foam increases. Micro foam must be prevented, as these micro bubbles will take a very long time to dissipate, thus negating all homogenization efforts.

As no solvents are involved, explosion-proof equipment is no longer required. For safe handling, please refer to the MSDS.

### Stirring time

Some THIXON™ & MEGUM™ containers come equipped with internal agitators. Stirring time will be dependent on the degree of settlement and on the stirring technique employed.

Flat stirrers, about half the diameter of the container, are usually more effective than propellers.

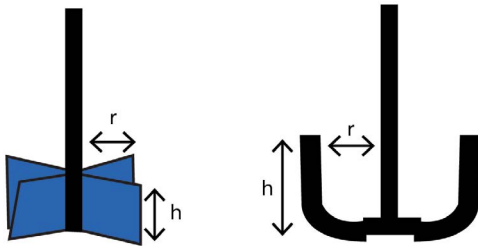
The stirring time should be long enough to obtain a homogeneous material. A rough guideline is provided in [Table 4](#) and [Picture 9](#), where stirrer geometry is correlated to the design and size of the container.

Agitation of the material through stirring should be performed at ambient temperature and should be continuous, whenever possible. In general, the stirring time needs to be adjusted if settlements are still found and not fully re-dispersed!

**Table 4: Stirring Recommendation as Function of Container Geometry and Size**

Container (kg)	r [%D]	h [%H]	rpm	time
1-5 sample	~ 30-50	~ 10	~ 20-30	min 15 min
10 pail	~ 30-50	~ 10	~ 20-30	min 30 min
25 pail	~ 30-50	~ 10-20	~ 20-30	min 60 min
200 drum	~ 40-75	~ 10-25	~ 40-50	8-24 h

(D=container diameter; H=container height)



**Picture 9:** Stirrer geometry recommended as function of container design and geometry

## Preparing MEGUM™ & THIXON™ for Application

### Dilution

MEGUM™ W-series bonding agents can be diluted to reduce their viscosity prior to spray or dip application. More specific information is available on the individual product technical data sheets.

These recommendations should be used as guidelines. The actual amount of diluent used should be adjusted to suit the application method and the equipment being used. Dilutions can be made by weight or volume, or to a specified viscosity (with less reproducible effects). For water-borne products, we recommend dilution to a specific density or dry solids instead.

Diluents should be added to the MEGUM™ W-series product and stirred to obtain a homogeneous mixture. The mixing operation should be carried out in a suitable area with equipment designed for safe use. Pigmented products should be stirred before use or dilution. Bonding agents and diluents should be at similar temperature to the working environment in which the coating operation takes place.

## Process control – viscosity measurement and controls

The viscosity of the water-borne bonding agents usually does not change significantly as a function of dry solids. Hence, when diluting, we recommend checking dry solids content or density instead. Especially for dipping processes, viscosity and dry solids should be checked regularly, at least at the beginning of the shift, if the process has already been established. When establishing a process, dry solids (which normally have been established during the pilot phase) should be checked several times during the shift, to gain information on the material's consumption and water evaporation. Especially with solids, any kind of contamination will falsify the dry solids measurement and should be prevented. In addition, the pH of the bonding agent used, at used dilution, needs to be kept stable within one pH value. A drift of pH will lead to coagulation and disposal of the material.

## Solids content – measurement and control

Dry-solids content determination is a very accurate method of monitoring dilution ratios and of ensuring that the bonding agent used is within the process specification set.

Determination of the dry-solids content involves evaporating the volatile solvent system from an accurately pre-weighed sample and then reweighing the resulting, dried solids residue. Solids content determination requires the use of a scale with an accuracy of 0.0001 g.

The dry-solids content, of the bonding agent under evaluation, is expressed as a percentage of the original weight of the product sample.

## Test procedure for determining dry-solids content

First, accurately weigh a suitable clean container. Disposable aluminum cups with a diameter of approximately 50 mm and a height of 12 mm are recommended. They should be clean and stored under dry conditions at ambient temperature. Then accurately weigh approximately 3g of the bonding agent into the solids-determination cup. Ensure that the sample has been properly stirred and is representative of the product-batch being assessed. Make sure that the weighing of the bonding agent proceeds quickly and accurately. Place the cup with the accurately weighed, wet bonding-agent sample into an air-drying oven set at 130°C. If the bonding agent has a ketone- or an alcohol-solvent base, it should be dried for two hours. If the bonding-agent solvent base is xylene, or one of the other relatively slow-evaporating solvents (water), the oven-drying cycle should be extended to three hours.

Finally, remove the dried bonding-agent sample from the oven and allow the sample to cool to ambient temperature in a dry area. Accurately reweigh the dried bonding-agent solids content sample cup. Calculate the percentage of dry-solids content as seen in Table 5a and 5b below. Two equation examples are given on the methodology. Please refer to the COA of the specific product to be used for the specific parameters.

Upon request, each batch of bonding agent is supplied with a certificate of analysis that gives details of its dry-solids content, viscosity, and density at the time of manufacture.

Table 5a Equation 1: Calculating dry-solids content

**For each sample taken:**

- $W_0$  • Weigh accurately two clean, dry aluminium foil cups
- $W_1$  • Weigh accurately apt. 3g of bonding agent sample into each cup.
  - Dry samples @ 130° C
    - 0,5h (THIXON and new MEGUM)
    - 2h (MEGUM Primers)
    - 3h (MEGUM Covers)
  - Allow dried samples to cool to RT
- $W_2$  • Reweigh dried solids ACCURATELY

• Solids Content =

$$DS[\%] = \frac{W_2 - W_0}{W_1 - W_0} \times 100[\%]$$

Weight of cup dry and empty

Weight of cup + 'wet' bonding agent

Weight of cup + 'dry' bonding agent

Table 5b Equation 2: Calculating coverage

$$Coverage = \frac{DS * 10}{s * DFT} = [m^2 / kg]$$

DS = dry-solids content [% by weight]

s = dry-film density in g/cm<sup>3</sup>

DFT = dry-film thickness in micrometer (μ)

## Film thickness – measurement and control

The thickness of the applied MEGUM™ or THIXON™ bonding-agent film can affect bond performance. Carefully monitoring and controlling applied film thickness, within the set specification, is recommended. The dry-film thickness (DFT) can be determined using non-destructive methods, like those used in the paint industry. Equipment is available for ferrous and non-ferrous metal substrates. DFT can be measured, for example, by IR heat-capacity determination via laser techniques, or with the well-known X-ray method, allowing the parallel investigation of primer and cover layers. Proper calibration of the equipment, using components with similar surface profiles to those being coated, is important to ensure consistent DFT determination results. The average DFT can be calculated using a minimum of five measurements.

An alternative method involves accurately weighing a component before and after coating, then using the surface area of the coated part together with the DFT of the bonding agent to calculate the applied average DFT.

## Coverage

Theoretically, MEGUM™ & THIXON™ bonding-agent coverage can be calculated using solids content, dry-film density, and DFT applied. This doesn't reflect the effect of overspray though, which is a function of the used application method.

Practical coverage is dependent on overspray and uniformity of coating; both are affected by the method of application. Coverage values can be used to estimate product usage and the economics as well as efficiency of the application method. For water-borne bonding agents, overspray is, by nature, reduced compared to solvent-based systems, as water has a higher density and tends to have a more accurate flight pattern of adhesive droplets.

For MEGUM™ water-borne water-borne systems, the following dry-film densities (calculated) are provided to calculate the coverage:

MEGUM™ W 9100	=>	1.6	g/cm <sup>3</sup>
MEGUM™ W 9200	=>	1.4	g/cm <sup>3</sup>
MEGUM™ W 9300	=>	1.7	g/cm <sup>3</sup>
MEGUM™ W 9301	=>	1.7	g/cm <sup>3</sup>
MEGUM™ W 9500	=>	1.5	g/cm <sup>3</sup>
MEGUM™ W 9400	=>	1.6	g/cm <sup>3</sup>

## Application Methods – Details

### Brushing

As water-borne bonding agents are low in viscosity and contain surfactants, brushing is NOT recommended due to the following reasons:

- Initiating foaming directly on the substrate
- Insufficient DFT control
- Puddling: causing thick DFT and lack of crack resistance

### Spraying

MEGUM™ W-series products are best applied by spraying which, in most cases, is possible without dilution in regard to cover and is best with dilution of the primer, if lower DFT (~5-10% with DI-water) is required.

In general, spray nozzles of 0.8-1.0 mm (31.5-39.4 mil) are suited perfectly, to apply MEGUM™ water-borne bonding agents. Please refer to our Technical Data Sheet for specific products to be used. Spray coating is used to apply MEGUM™ & THIXON™ bonding agents to large numbers of components or to large surface areas. With suitable jigs and masks, bonding agents can be applied to selected areas of a component. Application by spraying requires considerable technical control but will ensure a consistent, uniformly applied film. To ensure even coverage, parts should be rotated. Automatic conveyor-spray application machinery ('chain-on-edge') is used for maximum application efficiency. Hand-spray guns are useful for smaller runs. THIXON™ & MEGUM™ can be applied using the following types of spray equipment: air, airless, air mix, high-volume low-pressure (HVLP), or **electrostatic-spray** (all water-borne bonding agents are suitable for electrostatic application by nature).

Besides the general requirement of parts in touch with water (only plastic or SST to avoid corrosion), the usual spray equipment is suitable for water-borne bonding agents as well. The pumping system, however, needs to be approved for dispersion transport as described in paragraph "Application Generics."

One thing in common though, is the fact that dried films coagulate and cannot be re-dissolved. Therefore, flushing of the nozzle is essential as well as the addition of a particle filter right before the gun; otherwise, the nozzle will plug!

For using a spray-pot, for instance, at a single manual station, a split feed with DI water for downtimes of the spraying nozzle should be implemented. This allows a simultaneous flushing of the spray pistol during the shutdown process.

## Tumble spray (special spraying usually with lower required DFT)

Tumbling components in a rotating barrel containing MEGUM™ or THIXON™ bonding agents provides low-cost and uniform coverage. Best results are obtained by applying non-pigmented products to relatively small-sized components.

The coated components are dried by warm-air circulation in the tumbling barrel or after being discharged into drying trays.

Water-borne (pigmented) bonding agents are less suitable for regular tumble-spray application due to their requirement of relatively high energy input to flash off the water. This process may only work for small parts. However, specific application equipment, where heating/drying is done in different ways, is well known and proven for this application method. The higher dry-solids content of water-borne bonding agents is a definite advantage for this kind of application. (Please refer to your technical contact for more details).

The shape of the component and the effect of any abrasion between the components during tumbling can affect the uniformity of the applied bonding agent. In general, water-borne bonding agent films are less sensitive to this kind of interaction and concerns than their solvent-based counterparts.

## Dip coating

Dip coating is suitable for coating large numbers of components that require total coverage. The shape of the component is a major factor in determining whether it is suitable for dip coating. MEGUM™ & THIXON™ bonding-agent viscosity and the rate of withdrawal of the coated component from the dip tank are critical in avoiding excessive build-up on the edges of the component. Once set up, dip coating requires minimal supervision and maintenance. For small runs, hand-dipping is usually most convenient and requires no special equipment. When handling large numbers of components, a conveyor dip line may be used with in-line metal cleaning and drying sections.

For water-borne bonding agents, no specific requirements are requested, except that preheating the parts needs a temperature control of the tank, and the parts must not exceed 45-50°C.

If primer and cover are applied, a thorough drying after the primer application might be required before the cover application.

Water-borne bonding agents are suited for dipping applications and can be used either diluted or neat. All parts need to be corrosion proof if in contact with water. Like solvent-based bonding agents, these materials need to be stirred during application to prevent settling in the reservoir container. For dipping applications to provide consistent DFT, the withdrawing angle, as well as speed, have a significant influence. In general, when pulled out of the bath perpendicular to the surface of the tank, the thinnest DFT will be measured on the top and thickest on the bottom of the part.

## Critical

**Temperature:** The dipping tank needs to be equipped with temperature control and cooling to assure consistent ambient temperature, as the parts (which are preheated) will warm up the liquid over time.

**Skimming:** The surface needs to be kept moving to reduce a potential risk of skinning.

**Organic contaminants:** As water is incompatible with organic substances, any contamination tends to float on the surface. There is a great risk that these organic contaminants will coat the parts when transferred through the liquid's surface of the tank and prevent the primer from coating the same surface.

- It is strongly recommended to keep the tank covered when not in use
- Keep tank skimmed regularly to remove all potential organics
- Recirculate tank volume through a filter (100-250 micron) at least once a shift

## Roller coating

In general, roller coating is used to apply MEGUM™ & THIXON™ bonding agents to flat areas or onto cylindrical components. It is an efficient method to combine maximum coverage with minimum waste and uniformity of film thickness. Bonding agents are usually applied undiluted. Roller coating can also be used to coat cylindrical shafts or pipes using felt or foam rollers.

For water-borne bonding agents, this kind of application can be utilized as well. It is recommended to keep the DFT lower than 6-8 microns and preheat the parts. Hence, the reservoir must have a temperature control and cooling to make sure the temperature stays ambient when processing.

Depending on the product, we recommend referring to your technical service contact for guidance.

## Storage and handling of coated components

Components coated with water-borne bonding agents can be handled and treated in the same way as with solvent-based products. However, because of the difference in film formation, compared to solvent-based bonding agents, the toughness of water-borne bonding agent films provides less sensitivity to mechanical influences.

Coated components can be stored for several days or weeks before bonding without affecting bond performance, but they must be protected from contamination. Unprotected components are particularly vulnerable to contamination by mold-release spray, hydraulic oil, and dust. Please remember, prior to vulcanization, never to handle coated parts with bare hands, as fingerprints will contaminate the surface with oil and potentially influence the overall performance. This is true for both water-borne and solvent-based products.

Never keep coated parts in direct sunlight or UV influence during storage.



A temperature change, from a cold storage area to a relatively warm bonding area, can lead to atmospheric moisture condensing on the component bonding area and may result in poor bonding performance. This is less critical for water-borne bonding agents as they need to be preheated anyway.

Compared to solvent-based dried films, once water-borne systems have coagulated and dried, they cannot be re-dissolved or re-dispersed. Hence, they need to be removed mechanically. Here, brass equipment is strongly recommended. Wetting with water prevents dusting during this operation.

Dried bonding agents need to be treated the same way as solvent-based and considered a combustible. The dried waste needs to be disposed of appropriately. Make sure the dried material does not encounter open flames, sparks or excessive heat.

## Methods of Bonding

### In-mold vulcanization/bonding

Compared to solvent-based bonding agents, vulcanization parameters and systematic controls apply for water-borne bonding agents to the same extent. Most bonded components are manufactured by in-mold vulcanization/bonding methods using compression (CM), transfer (TM), or injection molding (IM). THIXON™ & MEGUM™ bonding-agent systems provide the following advantages for in-mold bonding:

- Excellent lay-over of coated components that can be bonded several days or even weeks after coating if they are protected from contamination during storage.
- Resistance to prebake, thus allowing a time delay between loading of the mold and closing of the press. Stability to prebake is an advantage in loading multi-cavity molds.
- Effective with both low-temperature/prolonged-cure time and high-temperature/short-cure time vulcanization cycles. The vulcanization temperature-time cycle may be set according to the requirements of the rubber compound.
- Uncured films are resistant to the various physical stresses encountered in the in-mold vulcanization/bonding process.
- Hot-tear bond strength of the bonding agent to rubber bond is excellent, resisting the bond stresses experienced during the removal of the freshly bonded component that is still at molding temperature.
- THIXON™ & MEGUM™ products create a high cross-linking density between adhesive- and substrate-layers, as well as the bonding-agent and rubber, which results in a high adhesive strength of the composite.
- THIXON™ bonding agents films have good thermal stability at molding temperatures of up to ~200 °C

### Pipe and tank lining

In this process, water-borne bonding agents provide a significant advantage, as explosion-proof equipment is not necessary. Water, used as the solvent, will provide additional EHS advantages for workers, while working in a closed compartment. However, drying of these adhesives will require a longer time in comparison to solvent-based materials.

THIXON™ & MEGUM™ bonding agents are used in the lining of pipes and tanks in combination with tacky adhesive “tie cements.” These provide the initial tack necessary, while the rubber lining is rolled on in the lay-up process. The bond is formed while the rubber lining is being vulcanized using hot air, hot water, and/or steam. Generally, bonding pressure at the vulcanization stage is low; therefore, it is very important that good initial contact is made between the uncured rubber sheet and the bonding agent tie cement during the lay-up procedure. Ambient temperature-cured rubber compounds with chemically-initiated cure systems can also be used with THIXON™ & MEGUM™ bonding agents. Rubber-lined tanks and pipes are used in the chemical and mineral industries where the resistance of rubber to abrasion and chemical attack is particularly important.

### Post-vulcanization bonding

The post-vulcanization (PV) bonding process can be used to bond cured rubbers to coated metal surfaces or to other cured rubber substrates. The bond is formed by applying temperature and pressure to the assembled parts.

The advantage of PV bonding is in reduced mold costs. Parts, typically produced with this technology, are bushes and mounts with concentric metal tubes where the inner tube is in-mold bonded and the outer is bonded using a PV-bonding technique.

In general, water-borne bonding agents behave similarly to their solvent-based counterparts. The preheating of parts during application applies as well. The toughness of the film with water-borne bonding agents will provide an advantage during the preparation process of the parts compared to the solvent-based, mainly Cover II films (for more details please refer to your technical contact).

## Influence of Vulcanizing Conditions

### PV bonding

The quality of the rubber compound should be strictly controlled. The age of the compound is important in that scorched or partly-cured compounds may not give optimal processing characteristics. This may affect bond performance.

### Vulcanizing temperature

Rubber-to-cement failure can occur if THIXON™ & MEGUM™ bonding agents are allowed to prebake or precure before molds fill and pressurization operations are complete. Prebake of coated components usually occurs when either the mold-loading operation is prolonged, or there is a delay between loading the mold and the subsequent introduction of the rubber, mold closing, and application of molding pressure. High-temperature IM operations are particularly susceptible if mold-loading delays occur. The problem can be overcome by reducing insert loading time, reducing mold temperatures, or using a mold-loading jig to speed up loading time.

THIXON™ & MEGUM™ bonding agents designed to give prolonged resistance to prebake also are available.

Rubber compound sheet used for CM should have a clean bonding surface. There should be no surface bloom of accelerators, curatives, plasticizers or other rubber chemicals that may interfere at the MEGUM™ or THIXON™ adhesive rubber-bonding interface and eventually lead to poor bonding performance. If rubber compound has been stored for long periods, it is advisable to pre-mill before molding to ensure proper incorporation of compounding ingredients and provide a fresh bonding surface.

### Bonding pressure

Good contact between the rubber compound and THIXON™ & MEGUM™ bonding agents is essential to unleash maximum bonding potential. Bonding pressure is required to achieve this contact.

- **In-mold bonding:** rubber-fill and pressurization should be completed as quickly as possible after the coated components have been loaded.
- **Roller and pipe covering:** bonding pressure is applied by tape wrapping un-vulcanized rubber covering, followed by steam autoclave curing.
- **Lay-up:** used for the lining of tanks, pipes, and other vessels, bonding pressure is minimal – and surface contact pressure is applied only during the application of the un-vulcanized rubber sheet. Tacky tie cements are used; usually manufactured by dissolving some of the un-vulcanized rubber sheet in an organic solvent. The tie cement is applied over standard THIXON™ & MEGUM™ bonding agent primer/cover-cement bonding systems.

### Mold design

Transfer and injection molds for rubber-bonded components should be designed to reduce the possibility of MEGUM™ & THIXON™ bonding agents being removed, or “swept,” from the coated substrate as the un-vulcanized rubber enters the mold. The un-vulcanized rubber is of relatively high viscosity and moving at considerable speed as it enters the mold. Mold-entry ports or pressure-relief holes should not be situated adjacent to the rubber/substrate bonding interface.

Molds involving the assembly of many parts should be designed to minimize assembly time and reduce the possibility of prebake of the coated substrate between mold-loading and mold-closure. Multi-cavity molds may require a loading-jig to reduce the mold-loading time of the coated components and avoid prebake.

## Summary of Using Water-borne Bonding Agents

In general, water-borne bonding agents are on par with their solvent-based counterparts regarding performance and film integrity. With our new generation of MEGUM™ W-series products, the performance is at the same level as that of solvent-based systems, not only under static but dynamically-stressed applications as well (please refer to your technical contact for more details).

Some differences do apply and need to be considered:

- a) **AVOID FREEZING!** Storage conditions should be 0°C (32°F) – 40°C (104°F)
- b) Significant advantage regarding EHS: No explosion-proof equipment necessary
- c) **NO PAINT SHAKER** for re-homogenization
- d) Refer to stirring recommendations for re-dispersion step before application: prevent foaming
- e) Dilution possible with DI-water
- f) Dilution control with determination of density or dry solids recommended (viscosity change insignificant)
- g) As water-borne dispersions are incompatible with organic contaminants, thorough cleaning of surfaces prior to application is required
- h) Parts need to be preheated before coating: 50°C – 80°C (~120°F-180°F)
- i) Conventional spray equipment possible: parts in contact made of plastic or SST
- j) Best results with HVLP guns
- k) Storage of coated parts similar to solvent-based bonding agents:
  - A void contamination and UV light
  - Storage up to four weeks without loss of activity possible (refer to your own testing)
  - Film integrity improved compared to most other solvent-based products
- l) Cleaning:
  - wet bonding agent films (clean with water)
  - dried films (only removable with mechanical effort sometimes with organic solvents)
  - dried films (if completely dry, needs to be considered as combustible Class II and disposed of accordingly)

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