ThreeBond
TECHNICAL NEWS

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Bolts Pre-Coated with a New Aqueous MEC Process (Acrylic and Epoxy Types)

Introduction

The MEC (Micro Encapsulated Coating) process for bolt pre-coating is a technology where reactive resins and other materials contained in microcapsules are used to coat threaded surfaces such as bolts, screws, plugs and piping to provide sealing, locking, lubrication, etc. MEC processing is widely used with anaerobic adhesive for threaded parts, and as is applied in advance it provides a huge advantage in quality and process control. Such features give it excellent workability for a proven track record of long-term use in construction and other industrial applications.

In a typical MEC process, an organic solvent or water is used as a solvent during the process of coating threaded surfaces with microcapsules. At ThreeBond, we have worked on and released aqueous types which are environmental friendly while additionally organic solvents are often restricted by REACH and VOC regulations. Aqueous technology was established for the functional surface of threaded parts. However, compared to organic solvent systems, it did not completely satisfy performance needs such as when these surfaces are subjected to load or compositional restrictions.

In this publication we will show how we solved issues found in previous aqueous MEC and introduce a new aqueous MEC which shows performance equal or higher to these of solvent type.

Hereafter, ThreeBond is abbreviated as TB.

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1. Background

1-1 REACH Regulations

The European Union (EU) began implementation of chemical substance registration and regulation (REACH: Registration, Evaluation, Authorization and Restriction of Chemicals) on June 1, 2007 to protect health and the environment from potential hazards caused by chemical substances. Substances considered to have potentially serious effects on the environment and health, such as those that are highly carcinogenic, mutagenic or toxic to reproductive systems, are classified as SVHC (substances of very high concern), and 73 substances had been classified as such as of December 19th, 2011.

Among organic solvents, tertiary candidates for inclusion as SVHC have been published, as well as substances that should be listed as such subject to approval, further strengthening regulation of organic solvents.

1-2 VOC Regulations

VOC (volatile organic compounds) is a general name for organic compounds that become gaseous when exposed to the air. They are widely used in paints, adhesives, solvents and inks, and contain a wide variety of substances, such as toluene, xylene and ethyl acetate. VOC are said to be a causative agent of suspended particulate matter (SPM), which exerts a negative impact on photochemical oxidants and the respiratory system.

To suppress VOC emissions, the Air Pollution Control Act was amended in May of 2004, the Air Pollution Control Act enforcement ordinance (cabinet order) and enforcement regulation (ministry order) were amended in May and June of 2005, respectively, and VOC emissions began in April of 2006.

At the ThreeBond Group Pre-Coated Bolt Factory, we act in accordance with Article 17-15 of the Air Pollution Control Act, which states we "...shall endeavor to further controls on the emission and dispersal of Volatile Organic Compounds by selecting products that use a smaller quantity of Volatile Organic Compounds".

2. MEC Processing

Simply stated, the MEC processing technique involves coating bolts and other threaded surfaces with adhesive components in a dry film state. In order to achieve our objective, we apply a microencapsulating technique to many of the ordinary reactive adhesive components. In this way, the microencapsulated adhesive components are stable before the MEC processed item is tightened or screwed in. However, once screwed in or tightened, the shear force applied causes the microcapsules to burst, releasing the adhesive components, which then mix with the curing agent. Next, a polymerization reaction occurs which creates a firm bond to keep the threaded part from loosening or falling out. At ThreeBond, we offer two main types of MEC processing products: acrylic and epoxy.

3. Acrylic MEC Processing

3-1 Solvent Acrylic MEC (TB2411, TB2403, etc.)

Solvent acrylic MEC is mainly composed of microencapsulated acrylic monomers, binders and fillers. For the solvent acrylic MEC microcapsules, we use gelatin for the wall membrane, and coacervation (phase separation in aqueous solutions) is employed for our microencapsulation technique.

Using gelatin has several merits. It ensures a microcapsule with a strong wall film and dry film threaded surface that is stable after MEC processing. Additionally, a wide range of particle sizes can be manufactured for use with a variety of threaded parts. Further adjusting the amount of acrylic monomer and wall film reduces residue produced when excess adhesive overflows while tightening a threaded part. However, gelatin is difficult to use in aqueous solvent because it swells in water and is susceptible to moisture damage.

3-2 Conventional Aqueous Acrylic MEC (TB2457, TB2475)

ThreeBond has been offering aqueous acrylic MEC that uses water as a diluent since around 1998. The aqueous acrylic MEC we now offer uses a technique to take the specially shaped acrylic monomer that is the main component and disperse it in water and applies a new microcapsule technology. These products are environmentally responsible, stable and easy to handle.

Unfortunately, conventional products have a comparatively soft dry film and are easy to cosmetically damage due to the special compounding techniques used. When this kind of cosmetic damage occurs, it can also lead to the coated film peeling off which means that they must be handled with care.

To counteract this, we have focused our microencapsulation technology development on improving aqueous processing so that can be utilized in the same way as solvent-based products.

3-3 New Aqueous Acrylic MEC (TB2458, TB2468, TB2478)

Our new aqueous acrylic MEC products utilize a newly developed special synthetic resin microcapsule for structural components that are equal to solvent acrylic MEC. This microcapsule boasts excellent moisture resistance that stands up even when the diluent is water.

In conventional synthetic resin microcapsules, the wall film is physically brittle and there is concern regarding both capsule stability and the limitations of use for certain sizes of threaded parts (especially encapsulation of large particles). To solve these issues we employed a special method to improve microcapsule flexibility and developed a brand new microencapsulation technique. Exploring techniques to combine curing agent, binder and filler (Fig. 1) outside of microcapsules as well as coating amounts on threaded parts, we were able to solve issues apparent in solvent acrylic and conventional aqueous MEC and create a product that performs as well as or even better than they do.

Our product lineup (Table 1) contains different levels of post-curing adhesion strength, weak: TB2458, medium: TB2468 and strong: TB2478.

There are also grades that leave almost zero residue after being screwed in due to effective application of synthetic resin in the microcapsule wall film.

We have created a product with functionality that matches or surpasses conventional solvent acrylic MEC for use with a wide range of threaded parts.

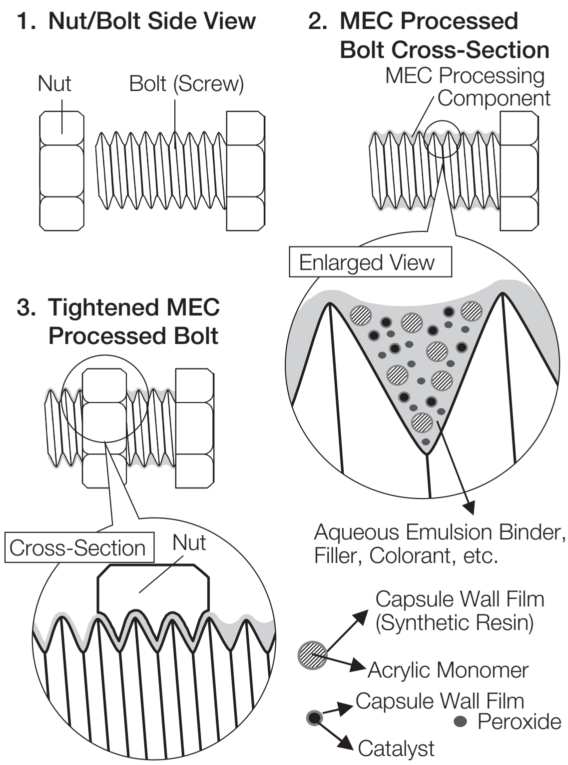

Fig. 1 New Aqueous Acrylic MEC Structure

●Storage Stability

Improved acrylic resin microcapsules are used to individually encapsulate acrylic monomers for storage stability superior to that of conventional products. As shown in Figure 2 below, stable adhesion is maintained over a long period of time in high temperatures and humilities more severe than typical environments.

Table 1 New Aqueous Acrylic MEC Product Lineup

Features		TB2458	TB2468	TB2478
Main Component		Methacrylic ester	Methacrylic ester	Methacrylic ester
Appearance		Green	Red	Blue
Adhesive Strength*1		39N·m	45N·m	52N·m
Heat Resistance Limit	Lock*2	100°C	100°C	130°C
	Seal*3	170°C	170°C	170°C
Curing Speed (20 to 25°C)	Practical Strength*4	30min. to 1hr.	30min. to 1hr.	30min. to 1hr.
	Final Strength	6hrs.	6hrs.	6hrs.
Minimum Applicable Screw Diameter		M3	M3	M3

*1. JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m, curing conditions: 25°C×24hrs.

*2. Temperature at which adhesive strength equal or greater than tightening torque can be achieved
JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m, curing conditions: 25°C×24hrs

*3. Pressure: Temperature without leakage at 10MPa
JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Attaching female screws on iron workpieces, tightening torque: 30N·m, curing conditions: 25°C×24hrs.
Sealant medium: Turbine oil

*4. Time at which adhesive strength equal or greater than 1/2 final strength can be achieved

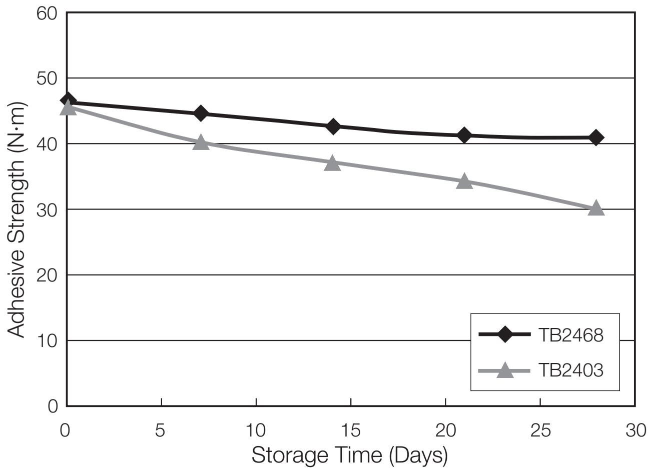

Fig. 2 Acrylic MEC Storage Stability

Testing method: Pre-coated bolts were stored in a 40°C, 95%RH environment for a set amount of time, then left to cool at room temperature before adhesive strength was measured.
Test piece: JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m, curing conditions: 25°C×24hrs.

●Axial Force

MEC processed bolts have the added characteristic of preventing leakage or slacking. As shown in Fig. 3 below, MEC processed bolts provide higher axial force and stronger fastening power than uncoated bolts. Axial force demonstrates tendencies similar to that of conventional products, so no significant changes are required in terms of handling tightening torque to make switching over to this product simple.

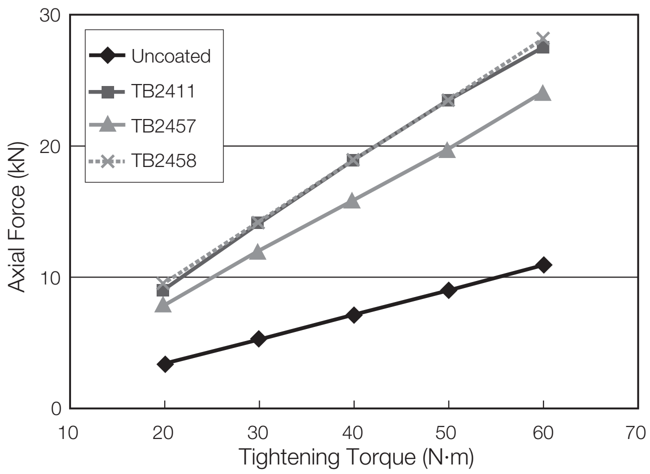

Fig. 3 Acrylic MEC Axial Force

Testing Method: Measured axial force at various tightening torques using a screwdriving tester with precoated bolts.
Test piece: JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tester: Screwdriving tester NST-500NM by Japan Instrumentation System Co., Ltd.

●Heat Resistance and Durability

Stable adhesive strength is maintained at 120°C (Fig. 4).

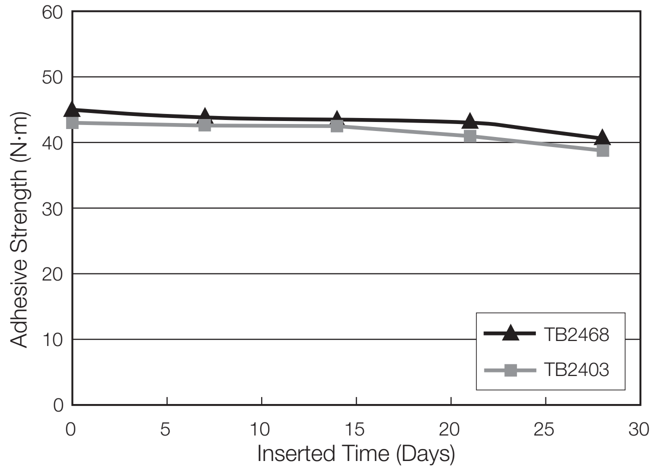

Fig. 4 Acrylic MEC Thermal Deterioration

Testing method: Pre-coated bolts tightened, cured and placed in a 120°C environment for a set amount of time, then left to cool at room temperature before adhesive strength was measured.
Test piece: JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m
Curing conditions: 25°C×24hrs.

4. Epoxy MEC Processing

4-1 Solvent Epoxy MEC (TB2430, TB2440B)

Solvent epoxy MEC is mainly composed of microencapsulated epoxy resin agents, amine curing agents, binders and fillers. Epoxy MEC demonstrates heat and chemical resistance that is superior to acrylic MEC, but has a slow curing speed.

Synthetic resin is the main wall film component of the microcapsules used for solvent epoxy. This process makes it possible to produce grain sizes smaller than in the capsules used for acrylic MEC to support compatibility with small screws, etc.

Because organic solvents were used in this solvent epoxy MEC, environmental concerns had to be taken into account.

4-2 Conventional Aqueous Epoxy MEC (TB2446, TB2446B)

In order to solve the issue presented by the use of organic solvent as a diluent in solvent epoxy MEC, we have developed an aqueous epoxy MEC utilizing special compounding techniques. The basic structure is the same as solvent epoxy MEC, composed of microencapsulated epoxy resin agents, amine curing agents, binders and fillers, with the same physical properties, such as chemical and physical resistance.

Table 2 Epoxy MEC Product Lineup

Features		TB2430	TB2448	TB2440B	TB2448B
Main Component		Epoxy resin	Epoxy resin	Epoxy resin	Epoxy resin
Diluent		Organic solvent	Water	Organic solvent	Water
Appearance		Blue	Blue	Orange	Orange
Adhesive Strength[*1]		70N·m	70N·m	64N·m	70N·m
Heat Resistance Limit	Lock[*2]	120°C	150°C	160°C	160°C
	Seal[*3]	170°C	170°C	170°C	170°C
Curing Speed (20 to 25°C)	Practical Strength[*4]	30min. to 1hr.	30min. to 1hr.	15hr. to 30hrs.	30min. to 1hr.
	Final Strength	24hrs.	24hrs.	72hrs.	24hrs.
Minimum Applicable Screw Diameter		M2	M2	M2	M2

*1. JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m
Curing conditions: 25°C×24hrs. (TB2448, TB2448B)
25°C×48hrs. (TB2430)
25°C×72hrs. (TB2440B)

*2. Temperature at which adhesive strength equal or greater than tightening torque can be achieved
JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m
Curing conditions: 25°C×24hrs. (TB2448, TB2448B)
25°C×48hrs. (TB2430)
25°C×72hrs. (TB2440B)

*3. Pressure: Temperature without leakage at 10MPa
JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Attaching female screws on iron workpieces, tightening torque: 30N·m
Sealant medium: Turbine oil
Curing conditions: 25°C×24hrs. (TB2448, TB2448B)
25°C×48hrs. (TB2430)
25°C×72hrs. (TB2440B)

*4. Time at which adhesive strength equal or greater than 1/2 final strength can be achieved

However, issues with curing speed still remained due to compositional limitations present in conventional aqueous epoxy MEC.

4-3 New Aqueous Epoxy MEC (TB2448, TB2448B)

The synthetic resin wall film of the microcapsule was made even more compact for the new aqueous epoxy MEC through unique technical improvements. This, in addition to individually encapsulating the main epoxy resin agents and curing agents sped up adhesiveness and enabled the creation of a product with excellent maintainability (Table 2).

●Basic Structure of TB2448 and TB2448B

The composition of the new aqueous epoxy MEC is shown in Fig. 5, below.

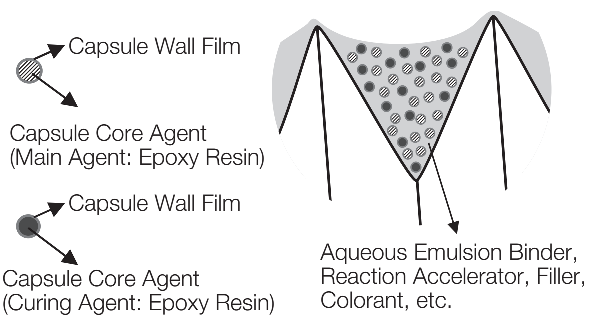

Fig. 5 New Aqueous Epoxy MEC Structure

●Curing Speed

For our newly developed aqueous epoxy MEC, elements are encapsulated in a different way than for conventional curing agents to successfully achieve a curing speed that surpasses conventional products and new aqueous acrylic MEC.

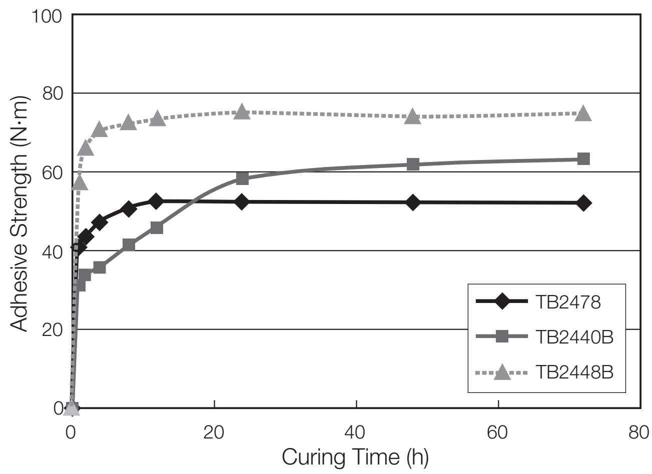

Fig. 6 Epoxy MEC Curing Speed

Testing method: Pre-coated bolts were tightened in a 25°C environment, then measured for adhesive strength after a set amount of time had passed.
Test piece: JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m

●Storage Stability

The newly developed, incredibly dense synthetic resin used for the wall film of the new aqueous epoxy MEC allows it to be stored at high temperature and humidity more severe than typical environments while suppressing reduction in adhesive strength (Fig. 7).

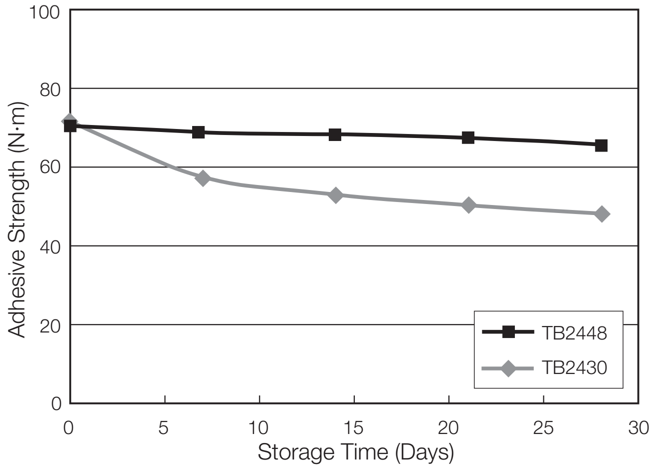

Fig. 7 Epoxy MEC Storage Stability

Testing method: Pre-coated bolts were stored in a 40°C, 95%RH environment for a set amount of time, then left to cool at room temperature before adhesive strength was measured.
Test piece: JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m
Curing conditions: 25°C×24hrs. (TB2448)
25°C×48hrs. (TB2430)

●Axial Force

Demonstrates axial force characteristics the same as in acrylic MEC described above (Fig. 8).

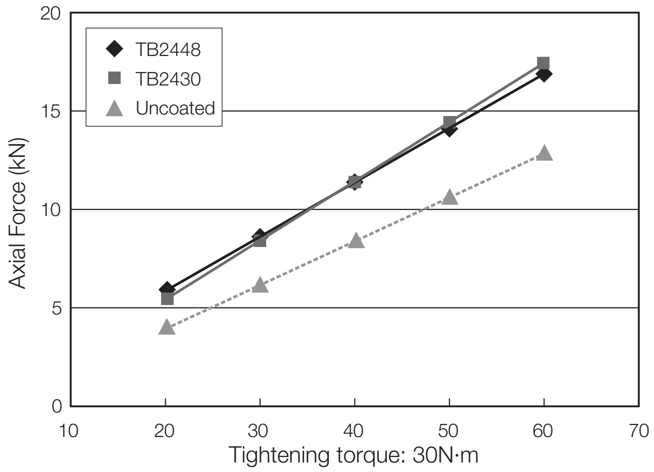

Fig. 8 Epoxy MEC Axial Force

Testing Method: Measured axial force at various tightening torques using a screwdriving tester with precoated bolts.
Test piece: JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tester: Screwdriving tester by Japan Instrumentation System Co., Ltd. NST-500NM

●Heat Resistance

One of the defining features of epoxy MEC is its incredible adhesive strength when hot as described in Fig. 9. Adhesion strength is high, even at high temperatures. (Temperature at which adhesive strength equal or greater than tightening torque that can be achieved is the lock heat resistance threshold.) Lock heat resistance threshold is 150°C for TB2448 and 160°C for TB2488B.

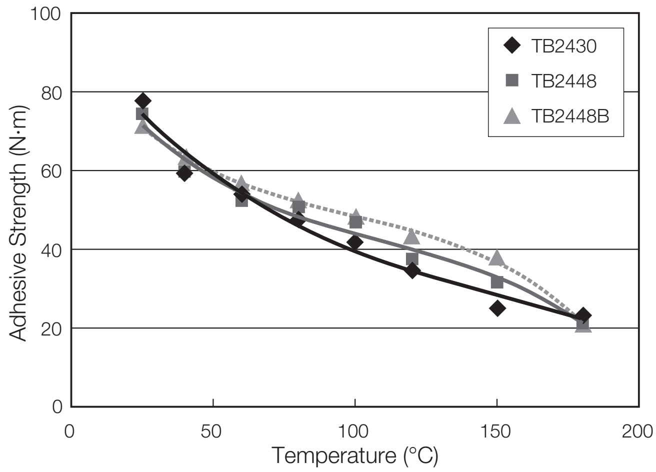

Fig. 9 Epoxy MEC Strength When Hot

Testing method: Pre-coated bolts tightened, cured and kept at various heats for two hours, then measured for adhesive strength at each temperature.
Test piece: JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque: 30N·m
Curing conditions: 25°C×24hrs. (TB2448, TB2448B)
25°C×48hrs. (TB2430)

●Heat Resistance and Durability

Stable adhesive strength is maintained at 150°C, as shown in the Figures 10 and 11 below.

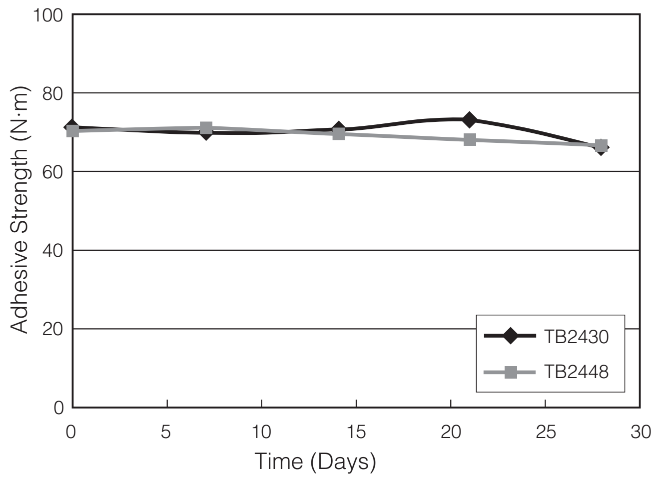

Fig. 10 Epoxy MEC Thermal Degradation (TB2430, TB2448)

Testing method: Pre-coated bolts tightened, cured and placed in a 150°C environment for a set amount of time, then left to cool at room temperature before adhesive strength was measured.

Test piece:	JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque:	30N·m
Curing conditions:	25°C×24hrs. (TB2448) 25°C×48hrs. (TB2430)

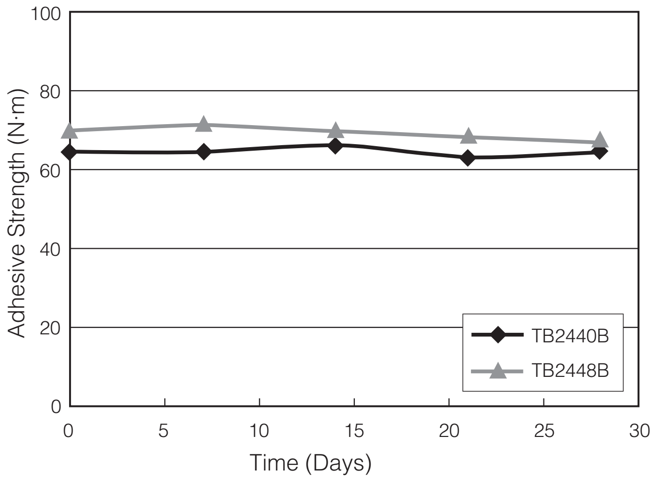

Fig. 11 Epoxy MEC Thermal Degradation (TB2440B, TB2448B)

Testing method:	Pre-coated bolts tightened, cured and placed in a 150°C environment for a set amount of time, then left to cool at room temperature before adhesive strength was measured.
Test piece:	JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Curing conditions:	25°C×24hrs. (TB2448B) 25°C×72hrs. (TB2440B)

●Chemical Resistance

Epoxy MEC demonstrates stability when exposed to various chemicals as indicated in the Figures 12 and 13 below. Use is stable in a wide range of applications.

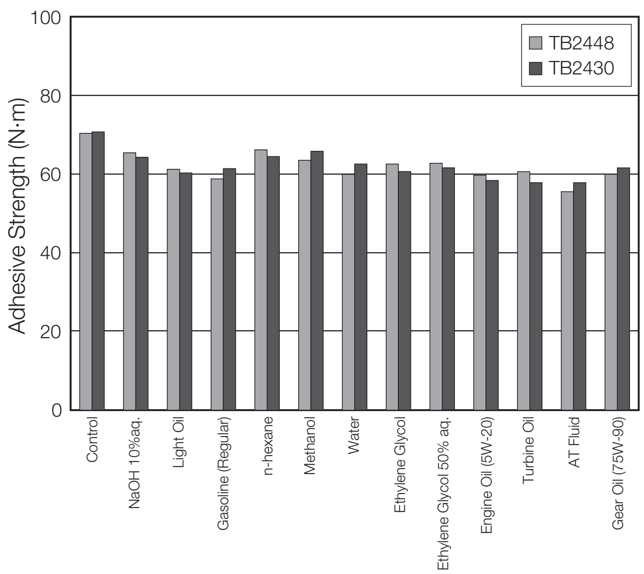

Fig. 12 Epoxy MEC Chemical Resistance (TB2430, TB2448)

Testing method:	Pre-coated bolts tightened, cured and placed in various chemicals. Once the set conditions have passed, the bolt is removed and measured for adhesive strength.
Test piece:	JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque:	30N·m
Curing conditions:	25°C×24hrs. (TB2448) 25°C×48hrs. (TB2430)

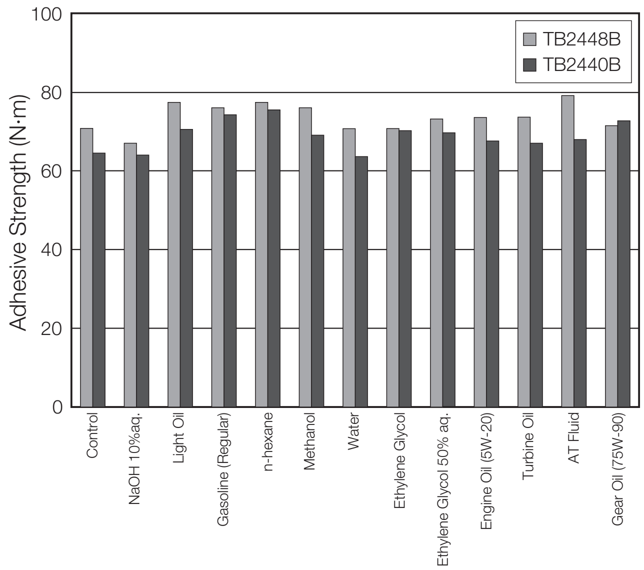

Fig. 13 Epoxy MEC Chemical Resistance (TB2440B, TB2448B)

Testing method:	Pre-coated bolts tightened, cured and placed in various chemicals. Once the set conditions have passed, the bolt is removed and measured for adhesive strength.
Test piece:	JIS class 2, M10×P1.5, zinc plating chromate treatment, hex bolt/nut
Tightening torque:	30N·m
Curing conditions:	25°C×24hrs. (TB2448) 25°C×72hrs. (TB2440B)

Closing

Our series of new aqueous MEC processed products have been praised for performance equal to or surpassing that of organic solvent products currently on the market. Through them, we now offer aqueous products that can be used under the same conditions as those using organic solvents.

Moving forward, we at ThreeBond will continue to develop products in line with laws, regulations and especially environmental concerns, both domestically and overseas.

<References>

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