EASTMAN

# Eastman™ CAB performance additive as a rheology modifier for anaerobic adhesives and sealants

Eastman™ cellulose esters (CAB, CAP, and CA) are versatile problem solvers and are typically used as binders, additives, film formers, or modifiers in many formulations for producing coatings, inks, adhesives, and other specialties. Eastman™ cellulose esters are based on up to sixty percent cellulose, one of the most abundant natural renewable resources. They also have FDA approvals and a non-hazardous rating to the German water classification (WGK).

In coatings, these high $T_g$ materials provide improvements in key properties such as rheology control, dry time, appearance, metal flake orientation, sag, flow and leveling, block resistance, and print resistance.

Fumed silica is used in many adhesives and sealants to control rheology. CAB can be used in conjunction with fumed silica to produce adhesives and sealants with unique rheology.

This technical tip demonstrates how CAB can alter the high and low shear rheology of a typical part A anaerobic pipe sealant formulation with no separation.

## What are anaerobic adhesives?

Anaerobic adhesives and sealants remain liquid in the presence of oxygen. When isolated from oxygen, for example, an anaerobic sealant or adhesive is sealed between a nut and a bolt on a threaded assembly. It rapidly cross-links or hardens to form a tough "cured" polymer that will bond to many metals. Typical applications for this product are in sealing of threaded connections against water, oil, gases, and most other materials transported through pipes.

A typical adhesive formulation is shown in this technical tip. The cure package for such an adhesive would contain cumene hydroperoxide or tert-butyl hydroperoxide in combination with an organic acid (maleic acid, saccharin) and a tertiary amine (n,n-dimethyl-p-toluidine or 1-acetyl-2-phenylhydrazine). Transition metal ions on the substrate surface play a key role in the cure chemistry, reacting with the hydroperoxide to generate free radicals. The redox decomposition of hydroperoxides by metal ions at room temperature produces free radicals, which, in the absence of oxygen, initiates polymerization. Anaerobic adhesives and sealants are sensitive to the type of substrate, requiring confinement on an active metal surface. For example, they cure rapidly on copper- and iron-rich surfaces, but cure slowly or not at all on cadmium or zinc metals. This difference in reactivity is due to differing reactivities of the various metals with hydroperoxides.

In the case of plastic substrates and non-reactive metals, primer compositions are sprayed or brushed on the substrate before bonding. The active ingredients in the primer provide the surface chemistry that initiates polymerisation. Solvent-soluble metal salts, such as copper naphthenate, are often sufficient for this purpose. The role of the maleic acid and saccharin is to provide soluble metal ions from the substrate surface which can subsequently decompose the hydroperoxide.

Table 1

## Anaerobic adhesive formulation

| Ingredients | No CAB (standard) | CAB 6.6% | CAB 13.25% |
|---|---|---|---|
| Poly(ethylene glycol) dimethacrylate functional monomer (*Aldrich*) | 67.15 | 62.70 | 58.25 |
| Aerosil™ 200 fumed silica (*Evonik Degussa*) | 3.75 | 3.50 | 3.25 |
| Eastman™ DBP plasticizer (dibutyl phthalate) | 19.30 | 18.02 | 16.75 |
| Zonyl™ MP1300 flouroadditive (*Dupont*) | 4.90 | 4.58 | 4.25 |
| Microthene™ FN51000 low density polyethylene (LDPE) (*Equistar*) | 4.90 | 4.58 | 4.25 |
| Eastman™ CAB 551-0.2 cellulose ester | 0.00 | 6.62 | 13.25 |
| Total | 100.00 | 100.00 | 100.00 |

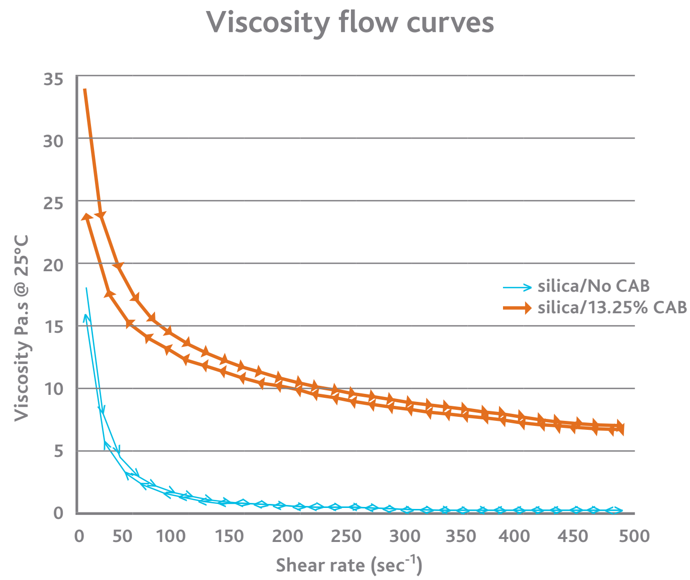
### *Formulation notes*

1. Eastman™ CAB cellulose ester was premixed in the Eastman™ DBP plasticizer before adding to the formulation.
2. To account for the extra addition of CAB, all of the ingredients in the formulation were reduced by the same proportion.
3. The formulations were prepared as follows
    a. The fumed silica was slowly added in several stages to the poly(ethylene glycol) dimethacrylate while mixing via a high-speed mixer over a 40-minute period at a speed of 4500 rpm. The high-speed mixer was stopped regularly to ensure that the temperature remained below 40°C.
    b. All the other ingredients were added in stages while mixing for an additional 40 minutes, ensuring the temperature remained below 40°C.

Figure 1

## Rheology (low to mid shear)

The rheology was measured for all three formulations. The flow curve of the standard fumed silica-containing system with no Eastman™ CAB cellulose ester is shown below compared with the fumed silica with 13.25% CAB.



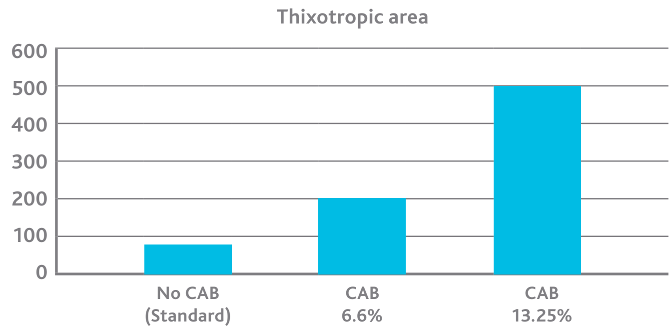

A Bohlin rheometer with a 40 mm, 1 degree cone geometry was utilized to conduct a continuous linear shear ramp from 0 to 500s-1 over 60s then back to 0s-1 over a further 60s in a controlled rate mode. A temperature of 25˚C with a 60s pre-shear delay at 0s-1 was employed and 30 sample points per minute were recorded giving a total of 60 measurements.

- The addition of Eastman™ CAB 551-0.2 cellulose ester produces an increase in thixotropy as shown by the increase in area between the flow curves. This increase in thixotropy should produce an anaerobic adhesive with increased flow after shearing.
- The addition of CAB 551-0.2 increases the viscosity at low and high shear rates. This enables the formulator greater flexibility in altering the rheology to provide the correct application performance as well as the final adhesive properties.

Figure 2

## Thixotropic area

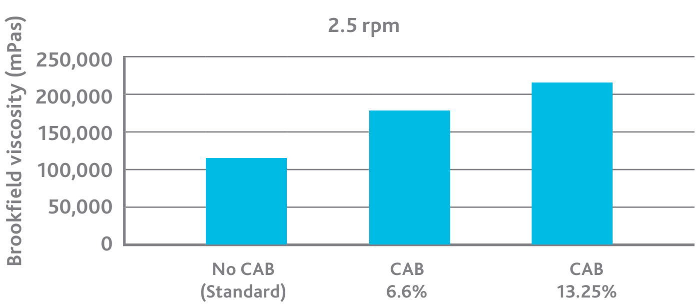
The thixotropic area of each of the three Bohlin viscosity curves was calculated and graphed below.




Eastman™ CAB cellulose ester increases the thixotropic area. As stated previously, this should produce anaerobic adhesives with increased flow after shearing.

Figure 3

## Level of viscosity at low shear Brookfield viscosity (mPa·s)



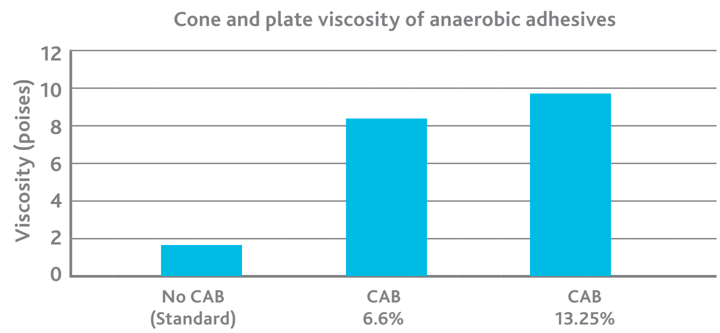

*Brookfield measurements at 23°C (spindles #3 to #6 @ 2.5 rpm–30 rpm)*

Adding Eastman™ CAB 551-0.2 cellulose ester increases the low shear Brookfield viscosity, enabling a formulator to adjust the rheology to produce a very high viscosity at low shear.

Figure 4

## Level of viscosity at very high shear ICI viscosity (poises)

Very high shear measurements were evaluated at 25°C on an ICI Cone and Plate (10,000 $sec^{-1}$). This shear rate could equate to the much higher shear rate produced when a nut is tightened onto a bolt.

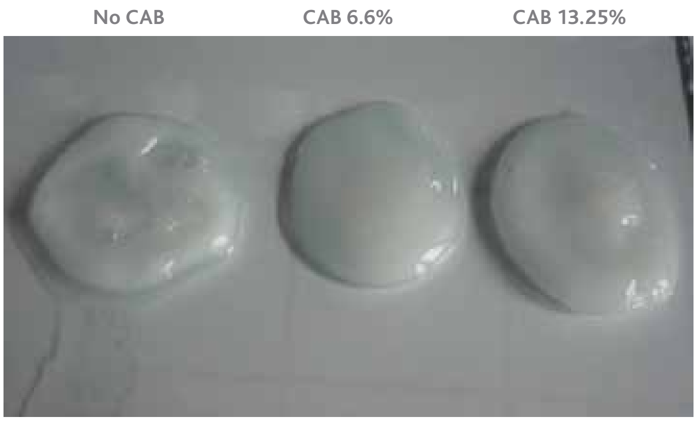



Adding Eastman™ CAB 551-0.2 cellulose ester increases the very high shear cone and plate viscosity. This may enable a formulator the option of producing an adhesive that has greater resistance to spreading after the tightening operations involved with threaded pipes, nuts and bolts.

Photo 1

## Separation

A small amount of sample was placed on a glass slide, and the amount of separation was recorded after an hour at ambient conditions.



The formulation with fumed silica alone produced separation of liquid component after storage at ambient conditions. This is clearly demonstrated on the glass panel above.

The rheology produced by the fumed silica and Eastman™ CAB cellulose ester combination was very good at preventing separation and syneresis of the formulation ingredients.

## Conclusion

Eastman™ cellulose esters should be considered important formulation tools for altering the rheology of an anaerobic adhesive formulation.

This rheological evaluation shows that in conjunction with fumed silica it is possible to increase the level of thixotropy as well as the low and high shear portions of the rheological profile.

Combining fumed silica and Eastman™ CAB 551-0.2 cellulose esters in an anaerobic adhesive formulation helps eliminate separation. No separation was seen in the combined fumed silica/CAB samples.

Such rheology flexibility could help formulators determine the best rheology for their adhesives and sealants which will ensure good application, performance, and storage stability.

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