ELVALOY™ Reactive Elastomeric Terpolymer (RET)

Lab screening guide

Suggested guidelines for initial screening of ELVALOY™ RET in asphalt for paving applications

DOW®

Introduction

ELVALOY™ Reactive Elastomeric Terpolymer (RET) differs from asphalt modifiers such as rubber, SBS or SB. To obtain the full benefits of ELVALOY™ RET, a reaction must occur between the terpolymer and the asphalt. Please read these instructions carefully and evaluate ELVALOY™ RET in a laboratory prior to any plant trials to determine the correct dosage and timing to complete the reaction.

There are two methods for completing the reaction of ELVALOY™ RET in asphalt:

1. The heat-reacted method to complete chemical bond between the terpolymer and the asphalt over a 12- to 48-hr period (depending on the asphalt binder).
2. A co-reactant technology to accelerate the reaction time to one to four hours.

It is important to follow the guidelines carefully.

Select proper base asphalt

The low-temperature properties – below approximately 4°C [40°F] – of a polymer-modified asphalt are determined mainly by the base asphalt. Select a base asphalt that meets the specified low-temperature requirements. The RET provides the high-temperature properties. ELVALOY™ RET does not normally change the cold temperature PG specifications.

The base asphalt must not be pre-acidified. ELVALOY™ added to acidified base will form gels as soon as it is added to the asphalt Those gelled particles will not be melted or dispersed with the asphaltenes in the binder.

ELVALOY™ RET grade selection and amount

The appropriate RET addition level depends on the base asphalt, the desired specification, and the RET grades chosen (consult with your Dow representative for more information). Typical terpolymer levels are between 0.5 to 2.0% by weight, but can be as high as 4%. Dow recommends initial evaluations at three terpolymer levels: 0.8%, 1.2%, and 1.8%. Results from these blend levels should provide information for fine-tuning the formulation.

While concentrated batches can be made, the amount of terpolymer that can be added to a batch is asphalt-dependent. Excess terpolymer levels can gel the asphalt binder and are asphalt dependent. Laboratory testing of concentrate blends must be completed prior to plant trials.

Typical laboratory equipment needed

A standard bench-top mixer (does not have to be high shear) capable of creating a small vortex as well as a hot plate or heating mantle capable of maintaining a constant temperature of 325°F or higher temperature are necessary. See Figure 1 for an example of such equipment.

Using the co-reactant technology

The use of a co-reactant, such as polyphosphoric acid (PPA) can significantly speed up the reaction between ELVALOY™ RET and the asphalt binder. The PPA is typically used at a concentration of 105%, however, concentrations greater than 105-115%. Typically 0.2-0.4% PPA by weight of the asphalt is used as a co-reactant. It is recommended that laboratory evaluations begin at 0.2% PPA and then increased to 0.4%.

Warning: It is critical that polyphosphoric acid be used as the co-reactant and not the more standard aqueous phosphoric acid (which is typically less than 85% in concentration). Aqueous phosphoric acid can flash off water causing the hot asphalt to spray out of the container. Please contact the PPA supplier for specific safety and handling recommendations.

Figure 1: Lab mixer and heating mantle

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Experimental blend preparation

1. Carefully heat the base asphalt sample in the original container in an oven until it is fluid enough to pour. An oven set at 165°C (325°F) should be sufficient.
2. Stir the sample until it is homogeneous before pouring into suitable blending containers. Quart and/or gallon paint cans work well.
3. Place the container of asphalt in a heating mantle or on a hot plate and heat to the desired blending temperature of 165-195°C (325-383°F). Once the sample is up to temperature, stir with a lab mixer set just fast enough to create a small vortex, without whipping excessive air into the sample (usually 200-300 rpm).

Note: ELVALOY™ RET chemically reacts with asphalt binder and the reaction is dependent on time, temperature, and chemical nature of the base asphalt. Some asphalts will oxidize or lose volatile components rapidly at high temperatures. The resulting increase in viscosity can overshadow the effects of the terpolymer and complicate the formulation process. Comparing formulation to control samples of unmodified asphalt is strongly recommended to determine the effects of the blending procedure on the asphalt.

4. Maintain the asphalt at the desired blending temperature for 10 minutes before adding the terpolymer.
5. Add the desired amount of ELVALOY™ RET, allowing the vortex to pull the pellets into the asphalt.
6. Continue to stir the blend for two hours after all the ELVALOY™ has been added. Check the blend periodically – the speed of the blender may have to be adjusted as the viscosity increases. Be sure to maintain the desired mix temperature. After two hours, the terpolymer should be fully dispersed and the heat reaction started.

Continue to step 7 or 7a to complete the laboratory screening:

- Go to step 7. for the heat-reacted method or
- Go to step 7a. for the co-reactant technology.

Note: During initial formulation, it is important to track the time of the reaction between the ELVALOY™ RET and asphalt by tracking the increase in viscosity – either absolute or Brookfield – or the DSR. Once the value stops increasing, the reaction is complete.

Procedure for heat-reacted method

7. Once the terpolymer has been mixed and agitated for two hours, take a sample of the asphalt and record the results (i.e., G*/sin δ or viscosity).
8. Place the container in an oven set to the desired curing temperature, normally around 165-195°C (325-383°F). Allow sufficient time for the reaction to take place. Cure times of 12 and 24 hours are recommended for initial testing. Record the G*/sin (δ) or viscosity every two hours until the stiffness has peaked and the reaction is complete.
9. After the reaction is complete, remove each container from the oven, run the desired tests. Use the data obtained to find the best cure time for the particular asphalt being tested. Often this cure time will be less than 24 hours, depending on the asphalt.
10. Once a target formulation has been developed, run all of the tests required by the specification.

Procedure for co-reactant technology

7a. Once the terpolymer has been mixed and agitated for two hours, the samples are ready for the co-reactant. For initial screenings, check that there is no gel formation present by adding 0.2 wt % (of the asphalt) PPA to the mixture and stir for 30 more minutes. Take a sample and test the viscosity or DSR.

Warning: Adding the acid too early in the process can prevent the ELVALOY™ RET from fully dissolving properly, and may cause lumps to form. If this happens, the ELVALOY™ RET must be mixed with the asphalt for a longer period of time prior to acid addition.

8a. Cover tightly with a lid and store in an oven at 165°C (330°F) overnight.

9a. After overnight storage, remove the sample from the oven and allow the container to cool for five to 10 minutes before opening carefully. Check for gel and run the desired tests. Use the data obtained to find the best ELVALOY™ level for the particular asphalt being tested.

10a. Once an initial target formulation has been developed, optimize the reaction by running screening tests using 0.2-0.4% polyphosphoric acid. Additional acid usually increases the elastic recovery but doesn't affect viscosity much.

11a. Once a target formulation has been developed, run all of the tests required by the specification.

Additional testing

After the formulation is developed and the necessary cure time has been determined, additional testing is recommended to anticipate and prevent manufacturing problems. Tests may include:

- Plant trials to decide how large-scale batches correlate with lab results.
- Stability tests to learn how the properties of the asphalt may change with storage time.
- Asphalt sensitivity studies to determine how variations in base asphalt properties, terpolymer level, storage temperatures, etc., may affect the final product properties.
- Asphalt compatibility studies to determine how to correct deficiencies in advance if off-spec product is made.
- Over-treatment tests to find out the maximum amount of terpolymer that can be added without forming an undesirable gel.

Warning: Heating asphalt in a sealed container can lead to pressure buildup inside the container, due to volatile components in the asphalt. Use caution when opening the container, a lid with a built-in valve can be used to manually relieve the pressure before opening the container.

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