DEVELOPING AT130, HIGH TEMPERATURE EPOXY GROUT

WHITEPAPER

ALPHATEC
grouting specialists

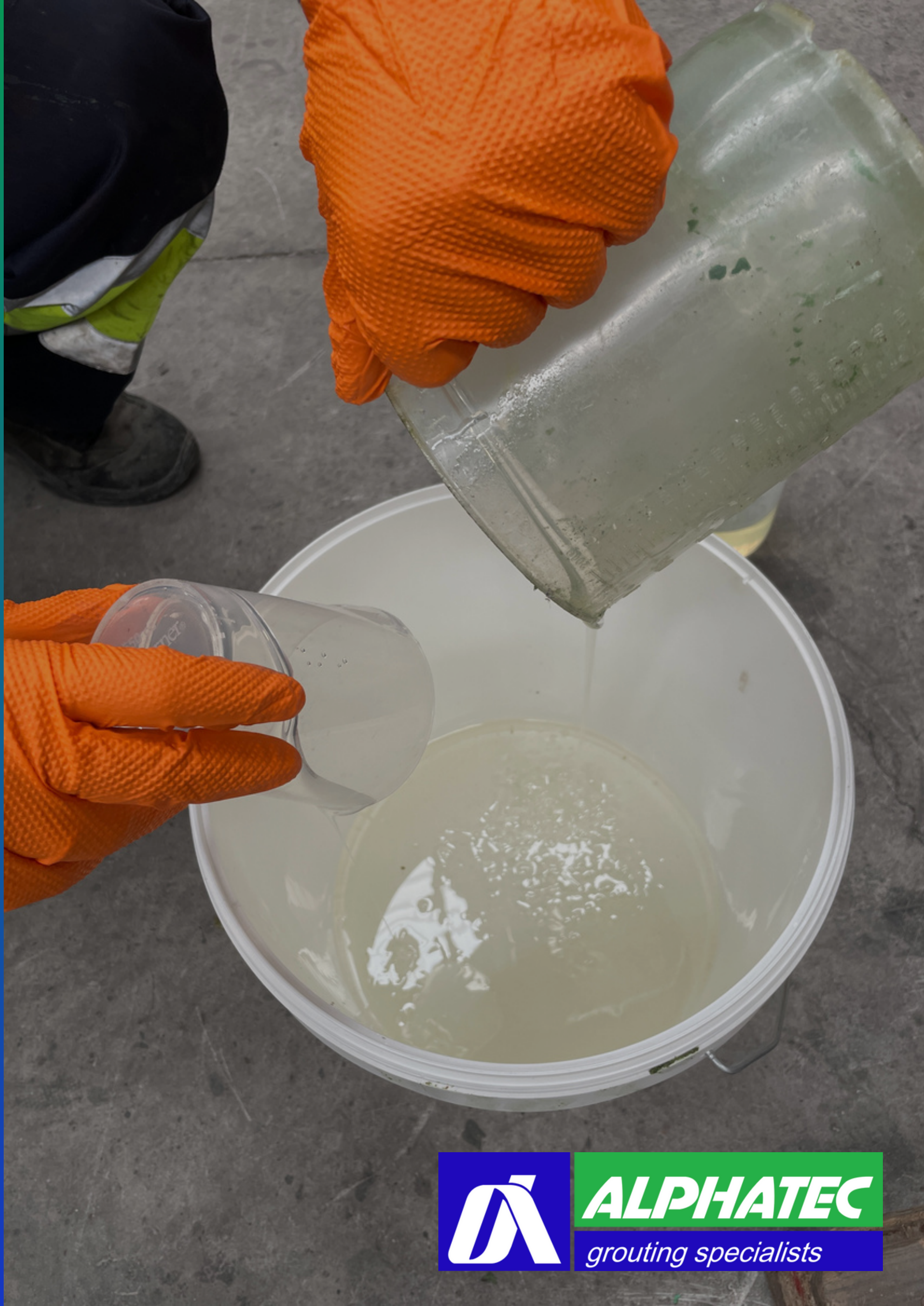
WWW.ALPHATEC-ENGINEERING.COM
INFO@ALPHATEC-ENGINEERING.COM

INTRODUCTION

Epoxy grouts are commonly used in high-temperature environments, but finding the right formulation that meets all the requirements of specific projects can be challenging. A high temperature epoxy grout should be able to:

- Withstand operating temperatures of about 150°C
- Tolerate aggressive, heat-driven chemical environments
- Have good mechanical properties, these include: good adhesion, high compressive strength, low shrinkage on cure
- Be pumpable whenever possible

In this white paper, we will discuss our journey to develop AT130, a high temperature epoxy grout that meets all these requirements.



BACKGROUND

The first step towards developing AT130 was to find a hardener that could withstand high temperatures.

After conducting extensive research, we set out to formulate AT 130 with amine-type hardeners, suitable for high temperature purposes.

We also acquired a peristaltic pump from Tomosada, Japan's finest, to conduct in-house pumping tests. And we collaborated with RDC, a reliable partner, to perform mechanical testing.

INGREDIENTS

To develop AT 130 as a 3K grout, we needed a suitable aggregate that could make it flowable. It needed to be easily available and readily mixable with green pigment. Silica sand turned out to be the best aggregate option because of its higher compressive strength, higher abrasion resistance, and improved thermal conductivity.

We also needed the right resin to impart flexibility and sturdiness to the grout. The options included:

- Bisphenol F
- Novolac-type
- Bisphenol A/F

STRATEGY – ROADMAP TO AT 130

We used a four-step strategy to develop AT130:

1. Work out the best liquid-to-hardener polymerization ratios. The higher the exothermic peak, the better
2. Try out aggregate-pigment combinations on said ratios
3. Mechanical testing on samples
4. Flowability testing on samples

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STEP ONE

The first step involved working out the best liquid-to-hardener polymerization ratios. We labelled each mixture and tested the exothermic peak of each ratio. We decided that the ones with higher T peaks were taken to the next stage.

Labelling	Liquid	Hardener	Volume ratio	Exothermic peak (°C)
AT 130 1	Bis A/F + Novolac	mixture	0.2	170°C
AT 130 2	Bis A/F + Bis F	mixture	0.2	170°C
AT 130 3	Bis A/F + Bis F	mixture	0.2	Below 100°C
AT 130 4	Bis A/F + Bis F	mixture	0.36	155°C
AT 130 5	Bis A/F + Bis F	mixture	0.2	125°C
AT 130 6	Bis A/F + Bis F	mixture	0.2	110°C
AT 130 7	Bis A/F + Bis F	mixture	0.2	128°C
AT 130 8	Bis A/F + Bis F	mixture	0.2	122°C
AT 130 9	Bis A/F + Bis F	mixture	0.2	160°C
AT 130 10	Bis F	mixture	0.36	Below 100°C
AT 130 11	Bis A/F + Bis F	mixture	0.2	160°C
AT 130 12	Bis F	mixture	0.36	175°C

STEP TWO

Next, we tried out aggregate-pigment combinations on the ratios that yielded higher T peaks.

We labelled each mixture and mixed it with either glass beads or silica sand to see which aggregate yielded the best workable mixture.

Labelling	Liquid	Glass beads?	Silica sand?
AT 130 1	Bis A/F + Novolac	Yes. Phase separation	Yes. Peak T: 45°C
AT 130 2	Bis A/F + Bis F	Yes. Phase separation	Yes. Peak T: 40°C
AT 130 4	Bis A/F + Bis F	Yes. Phase separation	Yes. Peak T: 25°C
AT 130 9	Bis A/F + Bis F	Yes. Foaming	Yes. Peak T: 30°C
AT 130 11	Bis A/F + Bis F	Yes. Foaming	Yes. Peak T: 30°C
AT 130 12	Bis F	Yes. Foaming	Yes. Peak T: 45°C

STEP THREE

We then carried out mechanical testing on samples using RDC's facilities. We labelled each mixture, added the appropriate aggregate and pigment, and measured the compressive strength of each sample.

Labelling	Liquid	Aggregate	Ratio (kg)	Pigment	Aggregate mixture	Compressive strength (Mpa)
AT 130 1	Bis A/F + Novolac	Silica sand type 1	1 : 0.6 (L) : 0.1 (H)	Green	-	69,6
AT 130 2	Bis A/F + Bis F	Silica sand type 2	1 : 0.6 (L) : 0.1 (H)	Green	-	80,3
AT 130 4	Bis A/F + Bis F	Silica sand type 1	1 : 0.6 (L) : 0.1 (H)	Green	-	43,8
AT 130 9	Bis A/F + Bis F	Sand mixture	1 : 0.6 (L) : 0.1 (H)	Green	Fine : Coarse	44,3
AT 130 11	Bis A/F + Bis F	Sand mixture	1 : 0.6 (L) : 0.1 (H)	Green	Fine : Coarse	57,7
AT 130 12	Bis F	Silica sand type 2	1 : 0.4 (L) : 0.12 (H)	Green	-	52,4

STEP FOUR

Finally, we conducted flowability testing on samples in-house using the Tomosada pump to ensure that AT130 was pumpable.

Labelling	Liquid	Aggregate	Compressive strength (Mpa)	Increasing ease of flowability
AT 130 1	Bis A/F + Novolac	Silica sand type 1	69,6	
AT 130 2	Bis A/F + Bis F	Silica sand type 2	80,3	
AT 130 4	Bis A/F + Bis F	Silica sand type 1	43,8	
AT 130 9	Bis A/F + Bis F	Sand mixture	44,3	
AT 130 11	Bis A/F + Bis F	Sand mixture	57,7	
AT 130 12	Bis F	Silica sand type 2	52,4	

RESULTS

After extensive testing, we found that the best formulation for AT130 was a Novolac-type resin or (Bis F). Silica and sand seemed to yield the best workable mixture, either as single-sized or as a mixture (RDC).

Mechanical testing showed that this formulation had a compressive strength of 80.3 MPa. Flexural strength measurements will follow suit. We also found that AT130 was pumpable using the Tomosada pump.

Labelling	Liquid	Aggregate	Compressive strength (Mpa)	Comments
AT 130 1	Bis A/F + Novolac	Silica sand type 1	69,6	Bottle neck: Novolac resin.
AT 130 2	Bis A/F + Bis F	Silica sand type 2	80,3	Bottle neck: Available in 10 days.
AT 130 4	Bis A/F + Bis F	Silica sand type 1	43,8	Bottle neck: Available in 10 days.
AT 130 9	Bis A/F + Bis F	Sand mixture	44,3	Sand mixture from RDC. Readily available.
AT 130 11	Bis A/F + Bis F	Sand mixture	57,7	Sand mixture from RDC. Readily available.
AT 130 12	Bis F	Silica sand type 2	52,4	Bottle neck: Available in 10 days

CONCLUSION

Developing a high temperature epoxy grout that can withstand aggressive, heat-driven chemical environments, has good mechanical properties, and is pumpable is no easy feat. However, by following our four-step strategy and conducting extensive testing, we were able to make advances in the development of AT130.

As we continue to develop AT130, we aim to address any potential challenges that may arise with our resin suppliers. By expanding our range of raw materials suppliers, we can increase our formulation options. Additionally, we plan to schedule mechanical testing at high temperatures with RDC.

FAQS

What is the meaning of "3K" in relation to grouts?

"3K" refers to a three-component material in which a liquid resin, a hardener, and an aggregate are mixed together to produce the desired grout. Similarly, "2K" denotes a two-component material.

What is the chemical reaction that takes place during epoxy resin polymerisation?

When an epoxy resin is mixed with an appropriate hardener, a 3D network of new chemical bonds forms among the reacting molecules. This creates a stable and durable solid, where the epoxy reactive groups on the resin chains are cross-linked together by the hardener.

What changes occur during cross-linking of epoxy resins?

The reaction generates significant heat, and the liquid or semi-liquid mixture becomes highly viscous, eventually hardening into a solid. Close attention must be paid to temperature changes and viscosity measurements.

FAQS

How is temperature measured during grout curing?
A special thermocouple is inserted into the curing mix to measure the temperature.

What is the typical compressive strength of Alphatec epoxy grout?
Our leading product, AT 800, has a compressive strength value of around 100 MPa, compared to the compressive strength of a typical cement grout which can be anything between 20 and 50 MPa.

Why is it essential to have multiple raw material suppliers?
In the case of epoxy grouts, the same chemical product can be marketed under different trade names or trademarks (be it an epoxy resin, a hardener, or an additive). Therefore, having access to the same raw materials from different suppliers is critical. This allows for more extensive formulation options and mitigates potential issues with a single supplier.