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TEST BEFORE YOU INVEST

COLOR, QUALITY, COST



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Fuller was formerly known as FLSmidth Cement

Test before you invest

FLSmidth experts describe the material testing necessary to determine if a particular clay is suitable to process into eco-friendly cement in terms of colour, quality and cost.

■ by **Necim Ourabah**, FLSmidth, Denmark, and **Steven Miller**, FLSmidth, USA

Clay is a naturally occurring material found almost everywhere around the world. Current standards allow for 50-55 per cent clinker substitution already, reducing process emissions by more than 40 per cent – a staggering achievement for an industry that is currently responsible for producing around seven per cent of global CO₂ emissions. Looking ahead, though, the potential is far greater. As practices evolve even further, performance-based approaches will likely gain wider acceptance such that 70 per cent substitution can become more commonplace. This, paired with other CO₂ reduction methods, will put the cement industry within sight of its net zero goals.

No wonder, then, that cement producers are increasingly interested in this eco alternative. But how do you know if your clay is the 'right' clay for the job? What guarantees should you get before you proceed with an investment? We called on FLSmidth's Mette Moesgaard, laboratory engineer, and Rasmus Franklin Momme, global chief process engineer, to tell us all about the company's clay testing

laboratory and pilot plant, which is used by companies from around the world to determine the likelihood of a project's success, based on the materials available and their properties.

Why clay matters

We can replace fossil fuels, we can increase energy efficiency, we can optimise every part of the process, and we still will not get close to a zero-carbon cement production process because of the embedded emissions that arise during the calcination of limestone. Carbon capture promises hope for the future, but that technology is not ready to take on this level of emissions just yet. Therefore, replacing clinker has become key to the cement industry's net zero vision. The less clinker we make, the less carbon emissions are released.

"Cement producers have long been replacing clinker in some quantities with things like limestone, fly ash and other

Testing, testing – how material and pilot-scale testing helps confirm clay suitability for eco cement



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pozzolans," explains Mr Franklin. "But clay offers a global opportunity for greater emissions reduction – first because it is so widely available, and second because of its pozzolanic properties after activation. However, not all clay is suitable to use as a clinker replacement. Which is where our laboratory and pilot plant come in."

Step one

Testing the raw clay

The first step is to evaluate if the clay raw material of interest (most often clays or shales) has the potential to be a suitable precursor for supplementary cementitious material (SCM) production. To start this evaluation process, the customer sends FLSmidth a 15kg sample of the material, which should be a representative sample from across the deposit. They typically send several different 15kg samples, enabling us to determine which ones have the most potential.

We use XRF, XRD and thermal analysis to determine the chemical and mineralogical composition of the sample and ask:

Dania – a decade of pilot-scale testing

Dania is one of the world's most advanced laboratories for the analysis of raw materials in cement production, receiving and testing tonnes of raw materials from all over the world.

The Dania pilot plant was commissioned in 2010 and reconfigured for SCM activation and colour control in 2020, but the site has been processing materials in one form or another since it was originally built as a cement plant in 1872.



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- Does it contain the desired minerals and at sufficient quantities?
- Are there any red flags in terms of emissions?
- What about the physical properties of the clay?

The physical properties are important because they will have an influence on wear and handling.

When we run these tests, we are looking at it from the perspective of 'is this feasible?' as well as 'is this financially viable?' For example, if the results were to show that emissions would be so high that the cost of air pollution control measures would outweigh the cost benefits of using calcined clay, then we would make the customer aware of that so they could decide whether or not to proceed.

"At this point, we can rule out any materials where the content of reactive clay minerals is too low," says Ms Moesgaard. "When that is the case, there is no benefit in continuing with the testing, and we would recommend the sender look for alternative sources."

The testing is not just a simple "pass" or "fail" but also provides insights to guide the project design. For example, information about the physical properties of the material feeds into project planning, so that equipment is designed to withstand highly abrasive or extremely sticky materials. Not only does this avoid costly pitfalls during commissioning, but it also makes the initial business case far more accurate.

Step two

SCM characterisation

"Provided the results from step one indicate that the clay may be a good match, we move on to step two," explains Ms Moesgaard. "At this stage, we calcine the clay in our laboratory (usually a sample size of about 2kg of material) and then evaluate the results. The calcination temperature will depend partly on the results of step one, but we may undertake calcination at up to three different temperatures to analyse the results."

Step two also includes an emissions analysis, an initial assessment of grinding behaviour, a characterisation of the calcined material, and what the material looks like after calcination. In addition, it includes an assessment of the potential to control the colour of the calcined clay, rheology and strength testing of a blended cement containing the calcined clay. In the case of rheology issues, we evaluate

Colour control

"The characteristics of the clay affect the colour, which is why colour measurements are important during our tests," explains Rasmus Franklin Momme, global chief process engineer, FLSmidth. "End users typically prefer grey cement, but adding calcined clay usually means the cement turns a shade of red, pink or brown. We measure the colour at the second stage of our process and undertake a laboratory scale colour reduction to understand what is needed to achieve the typical cement grey colour that customers prefer. We then apply our patent-pending colour manipulation process to determine the colour gradients achievable for the specific clay or cement mixtures applied to. This process will become even more important as cement manufacturers increase the quantity of calcined clay in the end product – the more clay in the mix, the more colour control is needed."

Without (left) and with (right) colour control

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optimisation by usage of superplastisers.

"The majority of the clays we have looked at so far have moderate or high potential to be used as SCMs," says Ms Moesgaard. "In the few samples we have seen where we have suggested not to proceed, one of them contained excessive pyrite, which would increase the sulphur emissions significantly, and necessitate a much higher capital investment. That is where the testing is so valuable. Pre-screening enables cement manufacturers to disqualify the worst materials – whether that is because the emissions are too high or the strength does not meet requirements."

FLSmidth's emissions assessment package includes:

- chloride (wet chemical method)
- fluoride
- sulphide
- total organic carbon (TOC)/organic carbon
- crystal water

- CO₂
- offgas test
- heavy metals screening, including Hg.

The characterisation of the calcined material includes:

- calcination degree based on thermal analyses
- distribution of minerals after calcination
- strength testing and calculation of strength activity index
- determination of water demand of blended cement mortar
- assessment of the potential to control the colour of the calcined clay, including colour measurement of calcined clay with and without colour control.

Step 3

Pilot-scale calcination and product evaluation

"We have a state-of-the-art pilot scale pyroprocessing facility at our R&D centre in Dania, Denmark," says Ms Moesgaard. "If

the material has successfully passed the first two steps of the process, we will undertake a pilot-scale calcination."

The installation includes:

- flash calciner string with temperature up to 1000 °C and a feed rate up to 100kg/h
- fluidised bed principle reduction vessel for colour control
- flash cooler string where temperature profiles are controlled – to ensure colour is preserved and heat recuperation will be applied in industrial configuration
- measure the full portfolio of gaseous emissions.

"To undertake the pilot calcination, we usually have the customer send us at least 3t of raw material," she explains. "We are aiming to produce as much of the finished product to send back to the customer as possible – usually at least a few hundred kilograms, sometimes more – so that they can perform their own large-scale tests on mortar and concrete mixes."

Having established the conditions for calcination in step two, in step three it is typically only necessary to produce the calcined material at one set of process conditions.

After dry crushing or grinding, the clay meal is calcined and then further tests are carried out to determine the degree of calcination, mineralogy after calcination and the colour. It is then ground to a standard fineness and tested for performance in a blended cement mix.

The mortar test of the final product includes one standard degree of substitution and the following tests:

- strength testing at one-, two-, seven- and 28 days according to EN norm, or one-, three-, seven- and 28 days according to the ASTM norm
- calculation of strength activity index
- water requirement to reach normal consistency of fresh mortar.

Gypsum is added to the blended cement to obtain an SO₃ content that matches the SO₃ content of the cement. During standard evaluation the lab uses a CEM I 52.5 cement as a reference, but it is also possible to include other cements or a clinker as part of the test.

"Step 3 is the time to experiment with process design, to define the optimum parameters for calcination, colour control and emissions," says Mr Franklin.



If the clay material has successfully passed the two first steps of the evaluation process, a pilot-scale calcination is undertaken

fourth step is really just to carry out any further experimentation the customer would like to try.

Whether or not producers proceed with the extra tests, they will receive all the information needed to decide whether to proceed with the investment. First, they will receive a complete summary and analysis of all testing performed, even guidance on the process design that would optimise their future operations. In addition, they will receive finished product (material), which they can use to carry out any further tests on their own.

"This work is so important to ensure our customers get the most from their investment. We usually invite customers to come to the plant at this stage, so they can see the results for themselves. It is really rare for a clay to 'fail out' of the process at this point, but it can happen."

Step 4

Extra testing

In step 3 we either use our own reference cement to mix up the blended cement for testing, or the customer can send us some of their cement products for us to test. This testing is usually to produce a binary (cement/calcined clay) cement, but sometimes customers also want to test other options, like a ternary cement comprising cement, calcined clay and limestone. FLSmidth offers both, so this

Conclusion

Calcined clay represents the best opportunity the cement industry has right now for large-scale emissions reduction, as well as reducing opex or even enabling a low-cost capacity increase. As standards evolve to enable greater use of SCMs, cement producers would be wise to seize that opportunity and position themselves for future changes to the cement mix.

However, while the market continues to demand grey cement, producers should also be aware that the more clay they add, the more colour control will be needed. To ensure the right quality and the correct colour, careful planning is required.

Pilot-scale testing enables cement producers to get the information they need to determine if their clay is suitable, and to design the process for the best results. ■

Reasons to use calcined clay

With LC³-50 (CEM II C/M) cement where the raw clay contained 15 per cent moisture, 10 per cent LOI (dihydroxylation), you can achieve:

- up to 40 per cent CO₂ emissions reduction per tonne of cement
- 30 per cent reduction in power consumption per tonne of cement
- 40 per cent reduction in fuel consumption per tonne of cement
- increase productivity without increasing emissions
- worldwide availability
- maintain cement quality with much-reduced environmental impact
- technology is proven and available now, with low ROI and low opex.

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