# New On-site Wet Milling Technology for the Preparation of Ultrafine Cement-based Grouts

Alex Naudts<sup>1</sup>, Eric Landry<sup>2</sup>

## Abstract

The use of ultrafine cement-based grout has been gaining importance for rock and soil grouting over the last few years. One of the more dramatic technical innovations in the grouting industry that has been sought is the ability to mill, on-site, a fine or ultrafine cement-based grout using regular cement, and locally available additives (fly-ash, pumice, slag, bentonite, clay, tailings, catalysts, sand). This paper presents the results of extensive research and testing program executed during the development of a mobile Particle Size Reduction Mill (PASREM).

The search for an inexpensive and effective milling machine for on-site production of ultrafine cement-based suspension grout has been ongoing since the early 1990's. The application of on-site milling would solve the problems associated with the use of classic pre-prepared ultrafine cement. This includes the agglomeration of particles that often results in a grout with a higher average particle size than desired, reducing penetrability of the grout, and the high cost of ultrafine cement.

On-site milling would also allow for the use of many locally available products and additives that could enhance the final grout and reduce the cost of producing the grout.

PASREM is a mobile milling machine used to produce ultra fine cements from readily available portland cements and additives. This process is preferably done by injecting a balanced cement-based suspension grout through the PASREM, immediately prior to grouting. This is further referred to as the wet milling process. The PASREM process can also be used to mill the aforementioned dry powders to microfine size, to produce a classic (bagged) microfine cement.

## The History of Wet Milling

The wet milling process is not a new concept. In 1992, Rodeo announced the first wet mill production process (Bosco et al., 1992) referred to as the Cemill<sup>R</sup> process. This process did not break through a number of problems: the coagulation of particles, rapid gelation of the grout, a sensitive and complex operation as well as some practical factors.

In 1993 Sandia National Laboratories also undertook the development of a wet milling process (Ahrens, 1993). This process was slow and cement particles began to coagulate and flocculate in the machine and the end product did not have the desired

<sup>&</sup>lt;sup>1</sup> President, ECO Grouting Specialists Ltd., 293199 8<sup>th</sup> Line, Grand Valley, ON, L0N 1G0; Tel. (519) 928-5949; anaudts@ecogrout.com

<sup>&</sup>lt;sup>2</sup> Senior Engineer, ECO Grouting Specialists Ltd., elandry@ecogrout.com

rheology (high viscosity and cohesion). This process turned out to be impractical for permeation grouting.

In 1999, a wet mill in Germany met criticism because the mill hardly reduced the particle size (Steiner, personal communication).

The complications associated with earlier attempts to develop a practical wet mill were addressed during the development of a milling machine described in this paper.

## Objective

The object of the program was to develop a feasible technology to produce fine and ultrafine cement-based suspension grouts via a wet milling and mixing process using cement. The main benefits of such an operation are the ability to custom mix and mill cement-based grouts to the desired particle size from regular cement, and locally available additives and admixtures.

The use of a wet milling process reduces the reliance on classic dry milled and bagged ultrafine cements that are expensive and generally available in developed countries or at a significantly increased cost in remote areas or underdeveloped countries. Regular cement is generally available everywhere. For many soil and structural permeation grouting projects applications however, regular cement is only marginally suitable for use. Especially where grout curtains for environmental applications are executed, the requirements for lower residual permeability have led to increased demand for improved mixing and fine and ultrafine cement-based grout.

In many grouting operations, the amenability of the formation to be grouted (Naudts, 1995) using regular cement-based suspension grout is not well known until the operation is well under way. It is therefore difficult to estimate the quantities of ultrafine cements that will be required. This leads to a compromise, in quality (allowing higher residual permeability in the rock grouting) when not enough ultrafine cement is ordered, or unnecessarily high costs when too much has been purchased.

Mixing and milling on-site suspension grout to the required particle size - only the quantities that are actually needed - has been the desire of those who want to democratise the grouting industry. Especially since the amenability theory quantifies the percentage of apertures accessible to water that are also accessible to a given grout (with particular rheology and particle size) it is possible to determine how fine the grout actually has to be in order to fill the fissures and pores of the medium to be treated.

For rock grouting, properly balanced, stable, cement-based suspension grout will penetrate clean apertures that are approximately two times larger than the largest particle size of the grout used. This means that properly formulated regular cement-based suspension will penetrate fractures as narrow as 90 - 100 micron (as was demonstrated in the Stripa Research Program). If ultrafine cement-based suspension grouts are used, the aperture width still accessible to these grouts is 20 - 30 micron. To put this in perspective: one fissure 80 microns wide (not injectable with regular cement-based grouts) per meter of borehole will produce a residual permeability of 3 x  $10^{-5}$  cm/s. By using ultrafine cement-based grouts the residual permeability of the formation can drop by a factor of 2 to 20 depending on the amenability of the formation for regular cement-based grout.

For soil grouting, on the other hand, soils with an in-situ hydraulic conductivity

value of 8 x  $10^{-2}$  cm/s can be permeated by regular cement-based grouts. Properly formulated ultrafine cement-based grouts (d<sub>95</sub><10 micron) will homogenously permeate soils with a permeability coefficient of 5 x  $10^{-3}$  cm/s over a radius of 0.80 m.

### **Research and Development**

The majority of problems encountered with grout prepared in wet milling machines were associated with the coagulation, flocculation and early gelation of the grout. It was felt that with the advancements in the field of grout admixtures that these problems could be overcome.

Liquid admixtures (viscosity, cohesion, and set modifiers) were introduced in the grout mix during milling using variable speed dosage pumps to each of the milling chambers. The rate of admixture injection could then be varied in each chamber to produce a grout with the desired rheology.

The second problem that plagued some early mills was the long milling time required to achieve a significant reduction in particle size. Initial research, therefore, focussed on the determining the most efficient milling process for reducing particles from an average size of 50 micron to as small as 3 micron in a short period of time. A number of field trips to were taken to observe various types of milling machines available in different industries. The machine selected for development was a mill with an upper chamber and a lower milling chamber. The chambers are filled with different types, sizes and volumes of grinding media. A diesel engine turns a shaft that causes the mill to oscillate and the grinding media to impact at the speed of sound. Adjusting the position of the shaft and the speed of rotation can vary the motion and size of oscillations of the mill.

During the R&D process, numerous modifications were tested including using different sizes of grinding media in each of the chambers, adjusting the volume grinding media in the chambers, changing the speed (rpm) of the mill, altering the shape of the oscillation curve, and the resident time of the grout in the mill. During the R&D process, specialists with extensive experience with a variety of milling machines assisted to determine the optimum parameters for particle size reduction. The information available from the mining industry was, at best, unreliable, and, at worst, conflicting. The research involved running numerous tests to establish the optimum configuration for the mill and to maximize efficiency by adjusting operating procedures. Samples of wet milled product were retrieved for each testing operation and sent to a laboratory for particle size analysis. Two particle size analysers capable of measuring a particle range of 1 micron to 100 micron were used for this research project.

After several months of testing, a system was developed to reduce the particle size of a regular cement-based suspension grout significantly while maintaining adequate production rates and desirable rheological characteristics. The best results were obtained when the grinding media in the upper chamber were of a larger size than those in the lower chamber. This produced a two-phase milling process. After extensive testing to optimise operational parameters on the mill, particle size analysis revealed that there was a dramatic reduction average particle size when all the milling parameters were "right-on". There was, however, always a faction of the particles that were deemed to be too large (12 - 18 micron range). It was determined that the resident time required to reduce the particle size of this faction would be detrimental to production rates.

Research into the use of separators, commonly used in other industries, produced a viable option for removing the larger faction of particles. The concept of further separating the larger faction of cement grout particles (as even regular cement-based has a fine faction) impacted on other characteristics of the grout. Several separators systems were tested to determine the optimum type necessary to remove the larger particles. The testing resulted in a device referred to as a 'PASREP', that uses two separators placed in series or used separately, depending on the reduction of particle size necessary for that particular application. The next step was to integrate the milling and separation process.

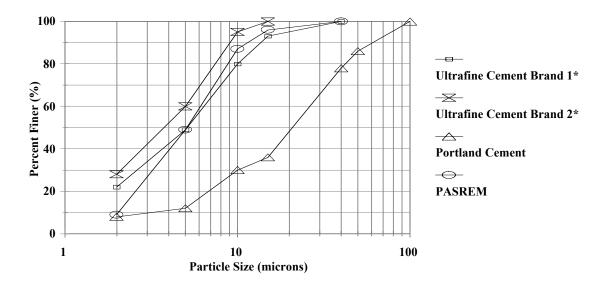


Figure 1. Particle size analysis of wet milled and classic ultrafine cement-based grouts

#### The Integrated Wet Milling and Separation Process

The production of fine and ultrafine cement-based suspension grout is a two-phase milling process followed by a separation process. The initial grout mix is produced in a paddle mixer, concrete truck or a high shear mixer containing water and any of a combination of regular cement, fly-ash (type F) pumice, slag, silica fume, bentonite, clay, retarders, superplasticizers and viscosity modifiers. The suspension grout is pumped into the upper chamber of the milling machine at a predefined pump rate. The suspension grout undergoes a first phase of milling in the upper chamber. The resident time of the upper chamber can be independently adjusted from the lower chamber. Admixtures are introduced during the milling process either into the upper and/or lower chambers. Just prior to the cement grout exiting the mill more admixtures can be added to modify the milled grout prior to use. If required, the milled grout is run through one or both sets of separators to remove the majority of the larger particles. The fine fraction is then pumped in an agitator tank for immediate use. The coarse faction is pumped back to the upper chamber of the mill. In this way, the milling time required to reduce the particle size of the larger faction does not impact on the overall production rate of the mill.

### Results

Figure 1 compares the particle size distribution of a regular cement-based grout, two ultrafine cement-based grouts prepared with commercially available ultrafine cements, and a wet milled cement-based grout prepared using regular cement. This illustrates the significant reduction in particle size between regular cement-based grout and regular cement-based grout after passing through the PASREM mill. A further reduction in particle size is achieved after the grout is passed through the separators. The two step wet milling process produces grout from regular cement that is comparable in particle size to grouts prepared using commercially available ultrafine cement.

By increasing the resident time of the grout in the mill the particles become finer. Since the permeability of a grout is predominantly dictated by its rheology, stability (resistance against pressure filtration) and cohesion, it is vitally important to place significant emphasis on these factors, in addition to the particle size distribution of the grout. In many instances, if the grout being injected into a formation has a favourable particle size distribution but poor resistance to pressure filtration, high bleed, and low cohesion, the end product will still be of poor quality. Freshly milled grout was injected into a series of identical sand columns (5% silt, 13 % moisture content) and the time to percolate through the column was measured. Although the test is far from ideal (Landry, et al, 2000), it provides a relative measure for the penetrability of a grout in a given medium. It was discovered that most commercially available ultrafine cement-based grouts, properly prepared in a high shear mixer, with the same water: powder ratio as the wet milled grout, did not permeate as quickly through the sand cylinders as the wet milled grouts. Although some of these commercially available ultrafine cements were verified to have a lower mean particle size and a finer d<sub>95</sub> than the wet milled grouts. In the opinion of the authors, the intense high shear mixing in the mill provides the explanation for the results.

Table 1 illustrates some of the grout properties of ultrafine grout produced in the PASREM mill. The wet milled cement grout was prepared using water, regular cement, slag bentonite, naphthalene sulphonate, and a retarder.

## Discussion

The demand for a workable wet milling process is best demonstrated by the fact that several others in the grouting industry have attempted or are still in the process of designing such a system.

For rock and structural grouting, the suitability of a given grout formulation is determined by its amenability coefficient. In essence, the initial permeability of the formation is determined via a water test (Lu<sub>water</sub> using Caron's equation) and in turn compared with one the permeability of the formation obtained using grout as a test fluid (Lu<sub>grout</sub>). If Lu<sub>water</sub> = Lu<sub>grout</sub> it means that all accessible apertures intersected by the borehole to water are also accessible to grout. This means that the particle size is adequate to pass through all the pores (soil) or fissures (rock). If Ac (Lu<sub>wa</sub>/Lu<sub>grout</sub>) is less than say 0.5 then less than 50 % of the apertures accessible by water are accessible to the grout. The means that a finer or more amenable grout is required to adequately treat the medium being grouted.

The need to mill the grout to a finer particle size is therefore governed by the amenability coefficient. The production rate and the need to use the separators follow from the aforementioned considerations.

Property	Standard or Test	Results
	Method	
Specific Gravity	API RP 13B-1	$1.42 \text{ g/cm}^3$
		(water/powder ratio: 1 to 1)
Bleed	ASTM C-940	0-4%
Initial Viscosity	API RP 13B-1	29-35 seconds
Pressure Filtration	API Filter Press	$45 \times 10^{-3} \text{ minute}^{-1/2}$
Cohesion	Lombardi Plate	1-3 Pascals – slow evolution in time until
		pump time has lapsed. Easy to adjust the
		viscosity by adding rheology modifiers during
		or after the milling process.
Initial Gelation	Wally Baker Shear Vane	adjustable between a few hours to 48 hours
Final Gelation	Wally Baker Shear Vane	adjustable between a few hours to 48 hours
Chemical Resistance		can be adjusted to enhance the chemical
		resistance against a variety of chemicals

Table 1. Wet milled ultrafine cement grout product data

Some drawbacks to the use of classic bagged ultrafine cement that the use of the wet milling process overcomes are:

• dry bagged ultrafine cement can cost up to 10 times more than regular cement;

• availability of ultrafine cements is limited and transportation costs from the manufacturers to more remote projects may be prohibitive;

• estimating grout volumes quantities required for a particular project can be difficult, leading to a shortage or excess of expensive utrafine cement;

• ultrafine cements have particle sizes 5 to 10 times smaller than regular cement with few options in between; and,

• dry bagged cements hydrate over time, causing coagulation of particles increasing the average particle size (i.e. they can have a short shelf life).

The cost of wet milling cement grout on-site is relative to the size of the project and the volume of milled product required. The cost of the mill would include mobilization and demobilization, operation of the engine and mill, and the cost of the cement and other additives and an operator. For small projects the costs of mobilization and demobilization typically cannot justify the use of a wet mill. As the size of the project increases, the relative cost saving of using a wet mill become apparent. The cash cost of producing, milling and mixing an ultrafine cement grout using regular cement is much lower than the cost of bagged ultrafine cement. By using the wet mill to produce fine or ultrafine cement grout from regular cement problems associated with shortages or oversupply of material are reduced since regular cement is usually readily available, and potential oversupply can easily be used for other construction applications.

## Conclusion

The nine month long, intensive research and development program succeeded in developing a wet mill capable of producing a balanced, stable fine cement-based suspension grouts with a significant reduction in particle size from regular cement-based grouts. The wet milling process not only produces better-mixed grout, it also reduces the particle size of the grout considerably. The milling process has been optimised; the size and shape of the milling media in the milling chambers, oscillation cycle and resident time and rpm have all been studied to improve efficiency. The wet milled grout has a better resistance against pressure filtration, lower cohesion, and hence better penetrability than grouts with comparable particle size distributions and composition that are prepared in a high shear mixer.

The wet milling process produces grouts with higher amenability for the formation to be treated, bridges the gap between regular and ultrafine cement and regular cement and makes grouting operations more economical as a result.

## Acknowledgements

The authors would like to thank Mr. Roger Willoughby of the National Research Council of Canada (NRC) for his assistance through this research and development project; and the NRC for its generous financial support.

## References

Ahrens. E. (1993). "Test Plan - Sealing of the Disturbed Rock Zone including Marker Bed 139 and the Overlying Halite below the Repository horizon at the Waste Isolation Pilot Plant." Internal publication, Sandia National Laboratories, Albuquerque, New Mexico.

Bosco, B., Bruce, D., A., De Paoli, B., and Granata, R. (1992). "Fundamental Observations on Cement-based Grouts (2): Microfine Cements and the Cemill<sup>R</sup> Process." Geotechnical Engineering Division, ASCE. New Orleans. pp. 486-489.

Landry, E., Lees. D, and Naudts, A. (2000). "New Developments in Rock and Soil Grouting: Design and Evaluation." *Geotechnical News*. September. pp. 38-44.

Naudts, A. (1995) "Grouting to Improve Foundation Soil." *Practical Foundation Engineering Handbook*. Ed. Robert Wade Brown. McGraw-Hill. pp. 5.277 - 5.400.

## **Related Material**

Heenan, D., and Naudts, A. (2000). "Advanced Grouting Program at Penn Forest Dam Results in Reduced Construction Costs and High Quality Product." *Geotechnical News*. June. pp. 43 - 48.