EGT Sampling Method; toward undisturbed sand sample

Jean-François St-Laurent
WSP Canada, Québec, Canada
Jean-Marie Konrad
Department of Civil Engineering, Laval University, Québec, Canada

ABSTRACT
Numerous publications showed and described the impact of sampling technique disturbance effects on cohesionless soil samples and behavior. This paper presents a new sampling technique for fine granular soils in a saturated state, called EGT sampling technique. The EGT sampling technique stabilizes the soil by using a gelatin solution. As the gelatin solution temperature decrease, hydrogenous bridges are created between gelatin molecules to form a dissolvable elastic gel. Undrained triaxial tests were conducted to establish the viability of the technique. Obtained results suggest that the developed method maintains the soil grain to grain structure. Observed divergence between behaviors are associated to system saturation difference. In order to support and assist the EGT technique, an advanced and unified thermo fluid-mass flow numerical model, based on 11 equations was developed. The finite element model, allowed determining the stabilized soil volume under various boundary conditions.

RÉSUMÉ
Tel que démontré et décrit par de nombreuses publications, l’échantillonnage des sols granulaires remanie l’échantillon, ce qui a un impact sur leur comportement géotechnique. Ce papier présente une nouvelle technique d’échantillonnage des sols pulvérulents fins saturés. La méthode d’échantillonnage EGT permet de stabiliser le sol par l’utilisation d’une solution de gélatine. Lorsque la température de la solution de gélatine diminue, des liens d’hydrogène se forment entre les molécules pour créer un gel élastique pouvant être dissous. Des essais triaxiaux en condition non drainée ont été réalisés afin d’établir la viabilité de cette technique. Les résultats obtenus suggèrent que cette technique ne modifie pas la structure du sol, que la configuration des contacts grains à grains est conservée. Les divergences de comportement observées sont reliées au degré de saturation du système. Afin de valider la méthode EGT, un modèle numérique d’éléments finis utilisant onze équations unifiant des principes thermique, d’écoulement et de transport de masse a été développé. Ce modèle numérique permet d’établir le volume de sol stabilisé par l’injection de la gélatine sous différentes conditions.

1 INTRODUCTION
It is well known that both static and cyclic shear strengths of cohesionless soils are highly influenced by grain to grain configuration, the mineral precipitation at grain contacts, the burial diagenesis, weathering and stress-strain history. In order to evaluate the in-situ strength and deformation characteristics of cohesionless soils, high quality in-situ tests or laboratory trials on undisturbed soil samples must be conducted (Yoshimi et al., 1978). Numerous sampling methods have been developed through the years to collect reliable samples for testing. As shown by Cuccovillo and Coop (1999), Vaid and al. (1999) and Hoeg and al. (2000) sampling method may impact the sample behavior by causing (i) water content (w) variations, (ii) changes in void ratio (e), (iii) chemical constituent variations, (iv) particles or constituents movements and mix and/or segregation. Studies agree that sampling damages the “brittle” structure of cohesionless soils.

To avoid the negative effects of sampling, indirect and in-situ methods were developed to evaluate geotechnical properties of soils deposits. Current practice is to link in-situ measurements to geotechnical properties through empirical correlations. However, as shown by Konrad (1990) those empirical correlations need to be validated with a highly accurate measurement of undisturbed soil/sample in-situ density.

The object of this paper is to introduce a new and promising sampling technique presenting high potential to be extremely efficient for obtaining undisturbed samples of saturated sand. This technique is referred to as EGT sampling (“Échantillonnage par Gel Thermo-fluidifiant” which loosely translates to “Sampling using Gelatin”). This sampling technique allows trimming and manipulation of samples without impacting soil mechanical behavior and density.

2 REVIEW OF CURRENT SAMPLING METHODS
Considerable efforts have been made in the past to develop and refine sampling techniques to obtain representative samples for laboratory testing. However, the most common sampling method is still the split-barel which provides highly disturbed samples.

Numerous studies indicated that large diameter tube samplers provide better quality samples (Lefebvre and Poulin, 1979). Currently, highly representative samples are obtained by in situ ground freezing using a large diameter tube sampler. This technique allows to increase the soil in-situ strength by freezing the inter-granular water. Hofmann et al. (2000) have shown that this sampling method is efficient when the freezing rate allows 9% of the porous water volume to drain away before freezing is completed. Moreover, it is recognized that the freezing technique works properly for materials having a low percentage of fines. Konrad and Pouliot (1997) indicated that disturbance of frozen samples due to thawing process can be limited by applying in-situ stresses to the sample before melting.
begins in laboratory. However, the in-situ ground freezing sampling technique is highly expensive. In order to reduce sampling cost, a confined cryogenisation sampling method was developed (Konrad et al., 1995). This technique consists of freezing the material located inside a large diameter sampler once the sampler is retrieved to the surface. This technique is less expensive than the in-situ ground freezing but produces frozen samples of lesser quality since mechanical disturbance occurred during sampler tube insertion into the soil deposit.

In conclusion, highly representative samples of cohesionless material can be obtained by freezing the pore water into the samples. However, it is realistic to believe that pore water density variation associated to the freezing or thawing process affects sample geo-mechanical behavior; even if the freezing rate is low and thaw is progressively applied in laboratory under in situ stress conditions.

3 LABORATORY DEVELOPMENT OF THE EGT SAMPLING METHOD

The EGT sampling method allows stabilizing and stiffening of fine cohesionless material without any pore fluid density variation. The solidification/stabilization process is obtained through the use of a solution of B-Type gelatin.

3.1 GELATIN DESCRIPTION AND CHARACTERISTICS

As described on www.gelatin.com, “gelatin is a purified protein, derived from the selective hydrolysis of collagen, the largest organic component of the bones and skins of mammals. The first English patent for gelatin production was granted in England in 1754 and is classified as a food ingredient. The most well known property of gelatin is its ability to form an elastic gel. Gelatin is composed of a unique sequence of amino acids: glycine, proline and hydroxyproline what provides the gelatin ability to form gels (figure 1). Being a polymer, gelatin’s macromolecular nature produces a viscous solution which at most temperatures and concentrations displays rheological properties that are Newtonian in nature. The viscosity characteristics of a gelatin type are primarily related to the gelatin molecular weight distribution. The viscosity of a gelatin solution increases with increasing concentration and with decreasing temperature. The rigidity of elastic gelatin gels increases with time as the gel matures, reaching equilibrium approximately after 18 hours of maturation (refer to figures 2 and 3). At the onset of gel formation, there is a tremendous increase in viscosity until the gel has completely formed. The strength of gelatin gels depends on the concentration and intrinsic strength (bloom) of the gelatin used which is a function of both structure and molecular weight. There are two different methods to obtain gelatin, an acid process (A-type gelatin) and an alkaline process (B-type gelatin). A-type gelatin is obtained by treating bones or skins in a vessel containing a dilute solution of acid. The B-type gelatin comes from de-mineralized bones soaked in a lime suspension or from skins soak in a caustic soda solution. At the end of the treatment, the raw material is washed thoroughly to remove any residual acid or alkaline solution.”
Literature review and laboratory trials allowed establishing that gelatin solution:

(i) is immiscible with water, allowing sample saturation with gelatin solution by injection process;
(ii) its setting point goes from 20°C and 30°C, and is function of the solution concentration. Over this temperature range, gelatin's state goes from a viscous liquid to an elastic gel as a result of hydrogenous bridge formation between amino acids (Science and technology of gelatin, 1914);
(iii) change of state is a progressive phenomenon that can be observed and identified through solution's dynamic viscosity increase as the solution's temperature decrease (figures 4 and 5)
(iv) when non oxidized, hydrogenous bridges creating the elastic gel, can be dissolved by increasing gel's temperature above the setting point temperature;
(v) Stabilized samples can be washed of the elastic gelatin gel by increasing the sample temperature and by injecting hot low vapor alkaline solution.

3.2 SAMPLE CONSTRUCTION PROCESS

In order to establish the viability of the proposed stabilisation technique, laboratory trials were performed. Uniform rounded Ottawa sand material was used to build samples in a triaxial apparatus using under water pluviation technique. Deaerated, distilled and chemically neutral water was used as saturation fluid during sample construction process. Samples were consolidated and densified and obtained void ratio (e) that varied from 0.67 to 0.77. The 15 tested samples height varied from 99.5 to 102.5 mm.

3.3 LABORATORY TESTING

The impact of the proposed stabilization process was studied by performing 15 undrained triaxial tests. Samples geomechanical behavior were studied in q-p', q-\varepsilon_a and \Delta u - \varepsilon_a domains. First the impact of the gelatin solution as samples saturation fluid was studied. Then, the geomechanical impact of gelatin washing and water re-saturation process were studied. Finally, triaxial testings were used to establish if stabilized samples may be manipulated and trimmed without geomechanical behavior modification.

3.3.1 REFERENCE SAMPLES

Five (5) reference samples were built with the under water pluviation technique and consolidated at desired densities. These samples were used to establish the standard geomechanical behavior of the used Ottawa sand. Obtained results allowed establishing a mean friction angle of the material at 31.15°. As shown below in figure 9, reference sample shown a contractive behavior up to ~0.75% of axial deformation and then shown a dilatancy behavior.

3.3.2 GELATIN’S SATURATED SAMPLES

Four (4) additional samples were built as described above, however to ensure complete water substitution and gelatin solution saturation, samples temperature was raised to 70°C and then a 70°C gelatin solution percolation was performed under a low hydraulic gradient (1 kPa/10 cm). To maintain the grain to grain original configuration and to limit fluid injection impact over the geomechanical behavior of samples, an horizontal constraint (\sigma_3') of 150 to 240 kPa and a porous back pressure of 180 to 190 kPa were applied during injection. Once the gelatin saturation completed, samples temperature was decreased to 10°C to allow a maturation time. After a maturation time of 16 to 18 hours, solidified samples temperature was increased to 70°C to allow gelatin gel to converts into the viscous solution and was used as samples saturation fluid. Samples temperature was maintained at 70°C during triaxial testing.

As shown in figure 6 and summarized in Table 1, samples having gelatin’s solution as saturation fluid do have a lower friction angle (29.44°) than reference samples (31.15°). Knowing that gelatin injection was achieved under a low hydraulic gradient and assuming that such injection
gradient could not lead to particles rearrangement under the applied confining pressure used during the injection process, it is believed that the gelatin solution acts as a grain to grain lubricator.

As shown by Khamehchiyan et al. (2007), pore fluid dynamic viscosity does influence soil behavior, what is in direct line with what was observed. Based on these results and observations, it was concluded that stabilized samples should be washed of the gelatin solution and saturated back with water prior testing.

### Table 1: Obtained friction angle from UIC triaxial assays

<table>
<thead>
<tr>
<th>Reference sample</th>
<th>Obtained friction angle</th>
<th>70°C gelatin solution saturated samples</th>
<th>Obtained friction angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ciusg2</td>
<td>31°</td>
<td>Ciuag3 29°</td>
<td></td>
</tr>
<tr>
<td>Ciusg3</td>
<td>30.75°</td>
<td>Ciuag4 30°</td>
<td></td>
</tr>
<tr>
<td>Ciusg4</td>
<td>31°</td>
<td>Ciuag5 29.25°</td>
<td></td>
</tr>
<tr>
<td>Ciusg5</td>
<td>31.5°</td>
<td>Ciuag6 29.5°</td>
<td></td>
</tr>
<tr>
<td>Ciusg6</td>
<td>31.15°</td>
<td>Mean 29.44°</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>Mean 29.44°</td>
<td></td>
</tr>
</tbody>
</table>

Figure 6: Impact of 70°C gelatin on the Ottawa soil friction angle

### 3.3.3 WASHED SAMPLES

Washing process consists of injecting 70°C deaerate distilled and chemically neutral water through 70°C samples that has been originally stabilized. The water injection was achieved under a low injection gradient, as per gelatin injection. To ensure complete gelatin washing, a low vapor pressure deaerated alkaline solution at a pH of 10.5 was also injected through samples prior being saturated back with deaerated distilled and chemically neutral water.

Undrained triaxial tests were undertaken upon three (3) washed samples. As shown in figure 7, the q-p’ behavior of washed samples against reference samples are highly comparable, suggesting that both samples friction angles are similar. Both samples stress-strain behaviors are similar as well (Figure 8). At the end of the contractile phase (εa~0.5%) or dilatancy phase (εa~2.3%) both samples showed identical q values. The observed divergence in the Δu – εa domain (figure 9) is associated to a lower saturation degree in washed samples.

Furthermore, as it can be observed in figure 10, initial stiffness of both types of samples, washed and reference, are similar. The observed divergence is associated to the washed sample longer consolidation period and samples saturation degree.

Performed testing program demonstrated that developed washing methodology allows samples to retrieved their initial friction angle and geotechnical behavior.

However, samples collected from field sampling have to be manipulated, cut and/or trim to be tested. In order to establish the impact of such manipulation, triaxial testing have being undertaken on sub-samples obtained from stabilized material as described within the following sections.

Figure 7: q-p’ behavior of washed vs reference sample

Figure 8: Stress-strain behavior of washed vs reference sample
3.3.4 WASHED SUB-SAMPLES

Two (2) sub-samples (50 mm of diameter and height) were obtained from 100 mm diameter and height stabilized samples. The manipulating and trimming work was performed as quickly as possible to limit gelatin oxidation. As it can be seen on figure 11, stabilized sand sample can be manipulated as if it was stiff clay. Recovered smaller samples were then installed back into the triaxial apparatus, washed and saturated back with deaerated distilled and chemically neutral water, as previously described. As shown in figure 12, the trimming and washing process did not significantly affect the sub-sample behavior, sub-sample and reference samples q-p' are similar. Figure 13 suggests that the stress-strain behavior of sub-sample is not affected when compared to the reference sample.

However, when compared to the washed sample, a significant behavior divergence is observed. It is assumed that the divergence is associated to the overall system saturation, which may differ from one trial to the other. In addition, the consolidation time is longer for sub-samples, and therefore may have impacted the samples behavior. Similar behavior divergence is observed in Δu - ε domain (Figure 14), which tends to endorse the above noted assumption. Additional testing should be performed to validate this assumption. Even though divergence is observed, it should be notice that samples stiffness are similar suggesting that grain to grain configuration has been maintained through manipulation process.
Figure 13: Stress-strain behavior of washed vs reference sample

Figure 14: Pore water pressure-strain behavior of washed and reference sample

3.4 NUMERICAL MODEL OF BOREHOLE INJECTION

Laboratory testing showed the viability of using gelatin as a stabilization method to maintain cohesionless soil geomechanical behavior. However, injecting such hot and highly viscous fluid into temperate soil presents numerous challenges.

A numerical model was then developed to simulate the gelatin solution. The finite element software FlexPDE was used to establish and predict the gelatin flow pattern and the amount of material to be stabilized under specific injection process. Spherical or axisymmetric flow created by an injection at the borehole end (Figure 15) was simplified through a 1D radial numerical model.

One of the key soil parameters that governs the fluid flow is the hydraulic conductivity (K). Saturated soil hydraulic conductivity is characterized by the soil physical characteristics and by porous fluid, as defined by equation 1.

\[
K = \frac{(\kappa \cdot \rho_f \cdot g)}{\eta}
\]  

Figure 15: Schematic representation of spherical or axisymmetric flow created during borehole end injection

Most of the time, it is assumed that both, fluid and soil temperature are similar. However, to ease and achieve proper gelatin solution injection, solution temperature should be elevated, around 70°C, which is higher than the soil temperature, estimated to be around 5 to 15 °C within depth of interest. To be able to model hot gelatin injection, the fluid and solid non thermal equilibrium model (Kim and Jang, 2002) has being used. This thermal model allows determination of the fluid temperature variation through time, which allows to establish the soil hydraulic conductivity variation through time as well, knowing the fluid dynamic viscosity variation with temperature (eq. 1).

Within the non-thermal equilibrium model, the solid temperature is established through the following equation:

\[
(1-n) \cdot \rho_s \cdot c_s \cdot (d(T_s)/dt) - (1-n) \cdot \lambda_s \cdot \text{div}(\text{grad}(T_s)) + h \cdot (T_s - T_f) = 0
\]  

Figure 15: Schematic representation of spherical or axisymmetric flow created during borehole end injection

and the fluid temperature through equation 4:

\[
n \cdot \rho_f \cdot c_f \cdot (d(T_f)/dt) + \rho_f \cdot c_f \cdot \nu_f \cdot (\text{grad}(T_f)) - n \cdot \lambda_f \cdot \text{div}(\text{grad}(T_f)) + h \cdot (T_f - T_s) = 0
\]  

where indices “f” and “s” refers to fluid and soil respectively. The temperature, density, porosity, thermal conductivity and specific thermal capacity are respectively:
T, \( \rho \), n, \( \lambda \), and c. The coefficient \( h \) is described by Dixon and Cresswell (1979) as:

\[ h = h_{sf} \cdot \alpha_{sf} \]

[5]

where \( h_{sf} \) correspond to the injected fluid specific surface described by Kuznetsov (1993) as :

\[ \alpha_{sf} = (6(1-n))/d_{50} \]

[6]

and \( h_{sf} \) is the coefficient of thermal transfer between the fluid and solid particules, described by Kuznetsov (1994) as:

\[ (1/h_{sf}) = (d_{50}/Nu_{fs} \cdot \lambda_d) + (d_{50}/\beta \cdot \lambda_s) \]

[7]

\( Nu_{fs} \) correspond to the fluid/solid Nusselt number, \( \beta = 10 \) for round particles, \( d_{50} \) = soil particle diameter of the passing 50%.

As any fluid, the gelatin flow into soil is governed by the following equation:

\[ \text{Div}(\text{grad}(h)) - \left(\frac{S}{T}\right) \frac{\partial h}{\partial t} = 0 \]

[8]

where \( S \) and \( T \) are respectively the storing and transmissivity coefficients of the soil and \( h \) represents the injection hydraulic head.

As mentioned previously, gelatin solution is immiscible with water. Considering that mass conservation principle can be applied, the amount of gelatin mass injected and transported through the soil by the advection process can be represented by the mass flux (Gelhar and Axness, 1983) represented by the following equation:

\[ J = J_a + J_{m_i} = (q^*C) - (n^*D_{m_i} \cdot \frac{\partial C}{\partial x}) \]

[9]

where \( J_a \) corresponds to the mass advection flux, \( J_{m_i} \) the mechanical dispersion flux. The injected solution concentration is represented by the C factor. The \( D_{m_i} \) factor represents the soil dispersivity and can be expressed as follow:

\[ D_{m_i} = \alpha_i^*v \]

[10]

where \( \alpha_i \) is the soil dispersivity; estimated by the \( d_{50} \) value of the soil. The parameter \( v \) corresponds to the fluid speed within soil’s pore:

\[ v = Q/(n^*A) = \frac{q}{n} = -\left( \frac{K}{n} \right)^i \]

[11]

As it can be noted, the fluid speed and subsequently the mass flow, is a function of the soil hydraulic conductivity (K) which is related to the fluid temperature (eq. 4) and injection gradient (i).

Assuming that the soil porosity is constant and uniform through time and space and that the injected fluid is non-compressible, the overall mass transport can be described as follow:

\[ \partial C/\partial t + v \cdot \text{grad}(C) - \text{div}(D_{ik}(\text{grad}(C))) = 0 \]

[12]

By coupling equations 3, 4, 8 and 12 together and by linking the soil hydraulic conductivity to the gelatin dynamic viscosity (figure 5), the developed numerical model allow to establish the gelatin solution distribution through time and space. By fixing a settling temperature as a threshold value, the developed numerical model allows to establish the amount of soil to be stabilized using a specific injection.

3.5 Laboratory Experimental set-up

In order to benchmark the developed model, obtained numerical results were compared to laboratory injection data. An injection cell was built to simulate spherical borehole injection process under a constant injection head. Thermistors were used to record temperature, allowing to determine the gelatin solution position within the domain. Figure 16 is a schematic representation of the laboratory setup used for injection simulation while Figure 17 is a photography of the experimental set-up.

Figure 16: Schematic representation of the laboratory setup used to simulate borehole injection

A 20 cm wide x 40 cm high plastic cylinder was filled with Ottawa sand using underwater pluviation technique. Samples void ratio (e) varied from 0.408 to 0.538. Samples boundary temperature was maintained constant with a cooling system as shown in figure 17. Hot gelatine injection was achieved through a 13 cm long plastic tube having a diameter of 2 cm. The injection point was ~5 cm below the ground surface, leading to an injection head (h) of ~ 8 cm.
Gelatin injection temperature was recorded at the control point TR. Temperatures recorded through thermistors T1 to T8 allowed to monitor, indirectly, the gelatin position. As per mentioned above, when the gelatin temperature decreases, the solution’s dynamic viscosity increases due to hydrogenous bridge formation between amino acid. Between 10 to 20°C the gelatine solution sets to an elastic gel, stabilizing the material. When gelatin seepage stopped by itself, meaning that the gelatin gel sat into the soil, a maturation time of 16 to 24 hours was applied prior stabilized bulb “excavation” to be performed.

Figure 18 shows a schematic representation of the gelatin laboratory injection process. As it can be seen, the limited injection depth led to a flow pattern modification. When the gelatine reached sample surface, the spherical flow pattern was modified to cylnindrical’s.

Figure 19 represents a stabilized bulb, obtained in laboratory. As it can be seen, the upper section of the bulb shows a cylindrical pattern compared to the lower section what is a nearly perfect sphere.

A total of 22 injections were achieved in laboratory under controlled and known conditions. The gelatin solution and soil temperature were modified to obtain stabilized bulbs of various dimensions. Stabilized bulb dimensions were then compared to numerically predicted bulb's dimensions under identical boundaries conditions and injection gradient. As shown at figure 20, the developed numerical model allows prediction of the stabilized bulb dimension under laboratory conditions within an acceptable precision. Most of the time, the numerical model was under estimating the bulb sizes by 5 to 15 %. It is judged that such precision is appropriate for the intended application. Observed divergence between the numerical model and laboratory data are associated to the soil’s intrinsic conductivity, storing and transmissivity coefficients estimation and to the limited depth of injection. These four parameters are affecting the gelatin flow pattern, misestimating one of them if affecting the numerical prediction.

**DISCUSSION AND CONCLUSION**
As demonstrated, the developed EGT sampling technique allows the manipulation of a fine granular sand sample as if it was a stiff clay material.

Larger stabilized sand samples could be trimmed and cut to smaller samples without any difficulties. Undrained triaxial testing programs showed that gelatin solution acts as lubricator when used as saturation fluid. However, a developed washing process allows the cleaning of stabilized sand samples from the gelatin solution and saturating it back with water. It has been shown that when such manipulations are properly achieved the stabilized sample geomechanical behaviors are not significantly affected. Moreover, the undrained triaxial testing program achieved over trimmed and cut sub-samples suggests that geomechanical behavior can be maintained. Performed testing program demonstrated that the EGT sampling method preserves samples friction angle and that samples stress-strain and pore water pressure-strain behavior were not significantly affected by the proposed stabilization and washing process. Both stabilized/washed and references samples stiffness into contraction phase were comparable. Results suggest that the sub-sample grain to grain structure has been preserve from manipulation remolding.

It is believed that observed divergence between behaviors is related to the testing system saturation degree difference and/or consolidation time longer for stabilized samples. Modified testing procedures should allow the convergence between both set of data to increase.

The developed numerical model, using the non-thermal equilibrium model between the fluid and the soil, mass and flow equations allows predicting, with enough precision the amount of material to be stabilized through a controlled gelatin injection process. The type, rate and temperature of injection are taken into consideration through the modeling.

However, the developed numerical model should be validated and improved by performing a field trial injection testing program. Such a trial should allow to establish the impact of groundwater flow on the gelatine flow pattern. In order to obtain a bigger stabilized bulb and to ensure that soil saturation is obtained, it is suggested to inject hot water prior to gelatin injection. Also, the injection rate must be establish based on field conditions to ensure that soil structure is not affected by fluid flow pressure. Additional testing should also be performed to: i) the impact of bacteria on the stabilization process (how long stabilized samples can be conserved), ii) what is the injection fluid pressure impact over the sample mechanical behavior, iii) what coring technique should be used, etc.

Additional studies and testing should be carried out prior to confirm without any doubts that the EGT sampling technique is providing real undisturbed cohesionless samples. However, on the basis of the above observations and laboratory investigations, it appears that the developed and proposed EGT sampling technique tends to be highly efficient to obtain high quality undisturbed saturated samples. Once fully developed, the EGT sampling technique should be an efficient, cheap and simple sampling technique, allowing to obtain highly representative fine cohesionless samples.

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