# UF grout system with new interactive chemical process

## Le systéme d'injection UF avec un nouveau procédé chimique interactif

A.V.SHROFF, Applied Mechanics Department, M.S.University of Baroda, Baroda, India
D.L.SHAH, Sardar Patel University, Vallabh Vidyanagar, India

SYNOPSIS: Injection of grout as strengthening and filler agent into alluvium and fissures or joints of natural rocks has been practicised by Civil and Petroleum engineers for several decades. The aim of this paper is to develop strong rigid UF gel by interactive chemical process of double order condensation with chelate complex, which can facilitates proper flow mechanism and can produce grouted sand with appropriate strength to resist foundation stresses and wash out forces. The scanning microscopy and Infra red spectroscopy have been employed to visualise a rigid unit cell for conceiving a theoretical model.

### 1 INTRODUCTION

While considering any foundation problem, a Civil engineer can examine use of grouting as one of the possible Geotechnical solutions. Broadly grouts are classified as particulate coarse grouts and watery low viscous fine grouts. The search is to understand the rheological behaviour of coarse and fine grouts. The vaious investigators have missed a motive to evolve a unified flow mechanism and strength interaction with natural formation. The flow mechanism and strength interaction of grout formation is mainly dependent on its time-viscosity relationship and strength. It is necessary to balance flow and strength behaviour of an injection mix within an ideal frame work to suit particular time-viscosity relationship and post gel strength which can resist foundation stresses and washout forces by proper occupation in spaces of formation. The microscopic changes associated with time-viscosity relationship of grout are important to correlate the limits of injectability and pumpability with respect to field condition.

This paper attempts to develope a UF gel by innovating a new interactive chemical process with the help of orgnic acid employing conceptual and equivalent weight approaches along with conventional frame work approach. The object is to evolve a rigid gel consisting of covalent and secondary bonds with chelate complex which can facilitate proper bonding and holding capacity with sand to be grouted. The appropriate time-viscosity and time-strength relationship are aimed to study the flow mechanism and strength interaction. The theoretical treatment is developed from a physical model conceived from a basic gellification mechanism supported by microscopy and Infra red spectroscopy.

### 2 GEL FORMATION AND ITS MECHANISM

UF gel is developed with the help of two component grout system from the following chemicals: (i) urea:formaldehyde (molar ratio 1:1), (ii) Catalyst/reagent:oxalic acid in presence of trace amount of melamine compound.

Oxalic acid interacts with urea formaldehyde in aquaous media dual way : (i) it provokes condensation reaction, (ii) it acts as a reagent to metalic salt of the alluvium to be grouted forming a 'Chelate complex'.

#### 2.1 Condensation reaction

The reaction of urea and formaldehyde in aquaous solution give first mono-methylol urea whose further reaction yields di-methylol urea. If the ratio of formaldehyde to amino-compound is 1:1, the initial reaction can lead to the formation of mono or di-methylol derivative rather than tetra. The addition of oxalic acid, provides H ion for the condensation and hydrolysis of mono, di or tetra methylol urea leading to the precipitation of insoluble compound methylene ureas. Further condensation of this by similar reaction lead to the final stage containing methylene linkage as shown in Infra red spectrum (Figure 1). This three dimensional net work structure by molecular diffusion grows out as a dense gel. The state of this reaction is controlled by the molecular ratio of formaldehyde to amino compound, pH, temperature and concentration of reactant. Melamine amino compounds produce a stronger hexamethylol derivative in comparison with mono, di or tetra methylol derivative of urea. Two stage condensation of derivative of melamine produce a three dimensional net work structure which have stronger branching and cross linked hexagonal polymer coils having arm length 2.5 micro metre, which are tied to each other via covalent bonds and secondary bonds in the boundry zones as shown in electron micrograph (Figure 2).

#### 2.2 Chelate complex formation

The characteristics features of such complex is the metal atom of alluvium which occupies a central position in chelate complex. It

| WAVE NO (cm$^{1}$) | INTERPRETATION |
|---|---|
| 770 | METHYLENE LINKAGE OR C H LINKAGE |
| 1000 | C-O STRETCHING |
| 1200 | C-O-E LINKAGE |
| 1400 | C=O BONDING |
| 1480 | C-O-C LINKAGE |
| 1680 | C O N $H_2$ GROUP |
| 3300 | HYDROGEN BONDING AMIDE LINKAGE N-H STRETCHING |

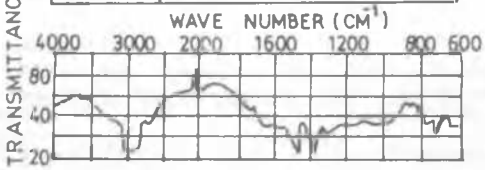
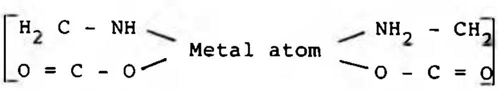



Figure 1. Infra red spectrum

2650X

Figure 2. Electron Micrograph

links to chelate complex by a covalent bond formed as a result of the metal atom accepting an electron pair from each non-metal atom of oxalic acid, later is called doner and the former is the acceptor atom. Alternatively, the oxalic non metal atom is the coordinating atom, therefore, it can also be called as coordinating bond. When metal atom is attached to more than one donor atom forming a hetrocyclic ring which is called 'Chelate ring' or 'Chelate complex' like a pointed horney nail and a molecule or ion from which it is formed is known as chelate agent. This chelate complexes are




entrapped in the cross link formation filling the spaces between the cross link and also they adhere the surface of the gel interacting firmly with their pointed horney nail with the pore spaces of the formation to be grouted, increasing the strength of the raw gel as well as the strength of the interacted grouted mass.

The gel occurs when the mix has pH 4.03 to 4.16 at the temperature of 27 degree centigrade, keeping gel time within 20 minutes having initial watery viscosity of 5 cp. During gellification, the colourless tansparent solution changes to milky white and finally gellified mass shows smoky white colour. Turbidity measurement during gellification help visual display of various gelation stages.

## 3 DEVELOPMENT OF GROUT MIX

Within the conventional frame work various concentration of urea formaldehyde in different proportion of acid are tried to get the desired rate of reaction and strength. Decrease or increase of oxalic acid concentration, retards or accelerates the reaction showing long and short gel time. The presence of melamine lengthen the gel time but increases the gel strength. The gel catalysed by oxalic acid in presence of melamine shows negligible syneresis. Increase of concentration of resin increases the strength, while increase of water dilution forms a weak gel. Ultrasonic study during the gellification process indicates the increase of shear midulus with time.

## 4 THEORETICAL TREATMENT

The conventional power model of Metzner and Reed (1955) do not follow the entire curve of time-viscosity of UF gel as the total behaviour from liquid to gel consist of various rheological changes.

$$\eta = S/(D)^n$$

Where, 'n' is degree of departure from Newtonian behaviour.

The mathematical model developed on the basis of the chemical process developed in this work supported by electron microscopy and X-ray differaction evidence of gellification stages of a liquid grout is

$$\eta = \exp[\,0.015\,(S)^{0.4}\,/\,D\,]$$

The vaccant lattice sites of a liquid grout filled up as the molecule grows with progress of time in a gradual fashion or instantly, the viscosity must necessarily mirror the change in free volume at any time.

## 5 RHEOLOGICAL ANALYSIS

Rheological analysis of developed grout system is carried out by time-viscosity study employing Brookfield Rheogram, USA. Shear rate - time (D-S-T) plot (Figure 3), shows Newtonian characteristics up to 5 minutes, thereafter it exhibits non-Newtonian quasi-viscous behaviour.

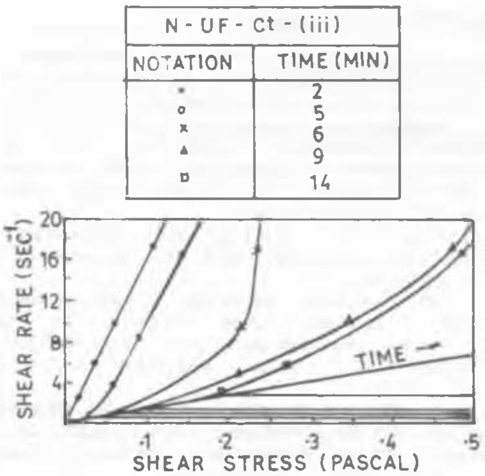



Figure 3. D-S-T plot

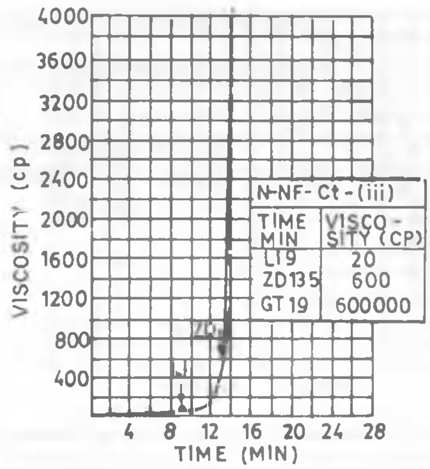



Figure 4. Time-Viscosity relationship

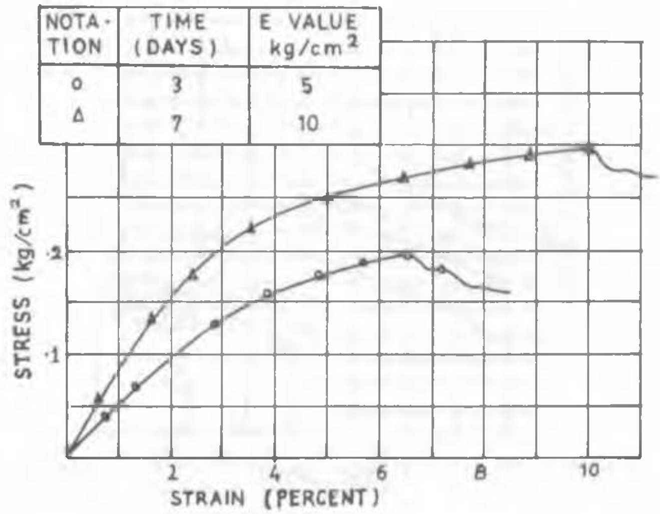



Figure 5. Stress-strain-time relationship - Raw gel

With progress of time, the quasi-viscous nature increases and tends to viscous behaviour promptly. The relative viscosity index as obtained from time-viscosity relationship (Figure 4) indicates that viscosity at zero displacement and gel time are 30 and 30,000 times the limiting injection time, showing final viscosity of 6,00,000 cp. The range of limiting injection time and gel time of 9 to 19 minutes indicates relatively quicker transformation from semi-viscous to viscous gel mass. Shear stress build up to limiting injection time is nominal compared to 645 pascal at gel mass. The rheological factor-time data exhibits rapid rate of acquirement of visco-elasticity after zero displacement time of 13.5 minutes. The ratio of viscosity to shear midulus deduced that after zero displacement time, grout becomes little elastic dominent rather than viscous dominent which tends to deviate computed viscosity to small extent from the mathematical model described in theoretical treatment. The deviation is of the order of 1.58 per cent. Strength-time data of raw gel as obtained from vane shear signifies a long term strength of 290 gm/sq.cm. Stress-strain time relationship (Figure 5) exhibits elastic nature up to 2 per cent and 1.75 per cent axial strains at 3 days and 7 days respectively. Stress-strain curve exhibits that the set mass becomes elasto plastic from elastic showing maximum peak value of 0.2 and 0.3 kg/sq.cm. at 3 and 7 days respectively. Ultimately failing to rupture. Failure axial strain increases with curing time.

In brief, in UF system the complete rheological changes from liquid to solid comprises of Newtonian to non-Newtonian, non-Newtonian to visco-elastic and visco-elastic to elastic.

## 6 GROUTED SAND MASS

Considering development stages and the rheological analysis, the appropriate grout is selected for injection purpose. Sand having D10 = 0.1 mm, specific surface area : 400 $cm^{-1}$, coefficient of permeability 2 x $10^{-4}$ cm/sec and uniformity coefficient of 2 is used for grouting. A grout mix is injected at a pressure of 2 kg/sq.cm in a steel mould of height to diameter ratio 2, and cured under moist condition.

### 6.1 Stress-strain characteristics

Stress-strain curve (Figure 6) of grouted sand sample indicates increase in peak stress with confining stress, reverts that strength-time relationship of UF grouted sand is strongly time dependent (Figure 7). Two categories of deformation are identified mainly time dependent linear elastic and time dependent

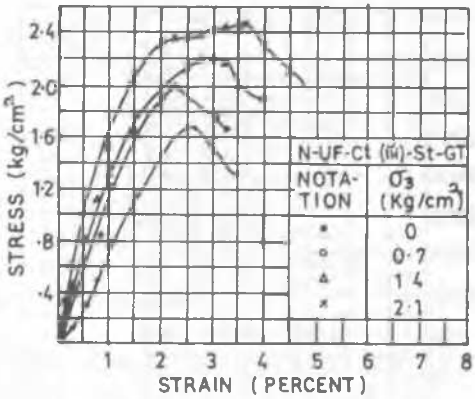



Figure 6. Stress--strain relationship - Grouted sand

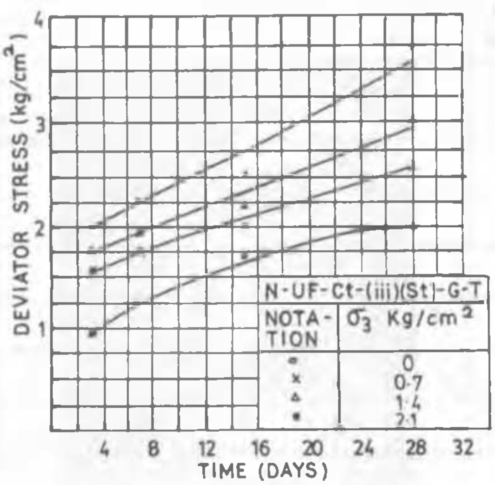



Figure 7. Strength-time relationship

non linear elasto plastic leading to rupture. E - value increases with time.

### 6.2 Permeability and adherent strength

After an aging period of 1 day followed by 4 days submergence, permeability reduction of 125 times in grouted sand is observed. Turbidity-hydraulic gradient relationship interprets that interacted grout resist critical hydraulic gradient of 85 showing adherent strength of 2.6 kg/sq.cm. Further, the washed out water from grouted sand shows more than 50 per cent survival of fish life, which is within prescribed standard for effluent disposal in river water. Also, leached out water gives COD and BOD within 250 and 300 values respectively.

## 7 CONCLUSIONS

1. Rate of reaction and gel strength are directly proportional to concentration of oxalic acid and urea-formaldehyde respectively. Initial Newtonian viscosity of UF grout facilitates injection even in fine sands and fine cracks of rock of size 0.1 mm or less, where cement or other high viscous chemicals are not workable.
The grout has controlled gellification time with gel stength and no syneresis and non-toxic nature.
2. In UF grout system, the complete rheological changes from liquid to solid consists of Newtonian to non-Newtonian, non-Newtonian to visco elastic and visco elastic to elastic.
3. UF grouted sand exhibits time dependent strength having two categories of deformation: (i) mainly time dependent linear elastic, and (ii) time dependent elasto-pastic leading to failure.
4. Mathematical model as suggested accounts all the rheological changes of UF grout system, which follows reasonably time-viscosity curve.
5. New interactive chemical process of double order condensation in presence of melamine and formation of chelate complex by metallic ion of alluvium to be grouted and non-metallic ion of oxalic acid help increase the strength of grouted mass.

## ACKNOWLEDGEMENT

This research work was performed at Geotechnical Laboratory, Applied Mechanics Department, Faculty of Technology and Engineering, M.S.University of Baroda, Gujarat, India. The financial support was obtained from the reasearch project of the department. Authors note the appreciation of Professor R.A. Vora of Applied Chemistry Department, M.S.University of Baroda, for his kind help in chemical analysis during the research work.

## REFERENCES

Caron, C. (1963). The developments of grouts for the injection of fine sands, grouts and drilling muds in engineering practice. Butterworth conf., London.

Clough, G.W. (1979). Silicate stabilized sands. Jr. of Geotech Engg. Div. Proc. ASCE, Vol.105, No GT-1, Jan. P. 65-70.

Metzner, A.B. and Reed, J. (1955). Flow of non-Newtonian fluids. A.I.Ch.E. Journal, 1, 434.

Shroff, A.V. & Shah, D.L. (1987). R E Grout system for improving fine sandy subsoils. VIII the Asian Regional Conference on Soil Mechanics and Foundation Engineering Kyoto, Japan.

Stetzler, B. (1982). Mechanical behaviour of silicate grouted soils. Conference on grouting in Geotechnical engineering, New Orleans, USA, P. 498.