INTERACTION BETWEEN SANDY DEPOSITS AND MULTIMOLECULAR ORGANIC SOLUTIONS BY STABILISATION OF SOIL

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Abstract. The paper presents the state of the art in grouting technologies for underpinning of old foundations, the analysis of grouting with multimolecular organic solutions and the results of investigations of dispersive soils stabilised with organic polymers over a period of one year. It describes the comparison between properties of unmodified carbamide resins and properties of modified carbamide resins. The following properties of resin solutions were investigated: density, viscosity, pH (alkalinity level) and evaporation of components from solution. The next stage of investigations involved the comparison of properties of alluvial medium dense sand stabilised with unmodified and modified resins.

Keywords: soil stabilisation, organic solutions, sand, grouting, foundations, injection, underpinning, environmental safety.

Introduction

Soil stabilisation by injections is an effective way to improve grounds under old buildings. A solid, water-proof artificial rock is formed by injecting low viscosity (similar to that of water) solution into the soil at low pressure (up to 1.0 MPa). While hardening, the solution forms crystal connections between disperse particles. The method resembles a natural process when free-flowing sand can turn into sandstone as it is cemented by natural cements.

Injection of stabilising solution into soil pores can be compared to injection of medicines into a human body. In both cases, the structure of the soil and a human tissue cannot be destroyed. The solution is injected at the lowest possible pressure and does its work as soon as it fills the pores, namely, cures the patient or reinforces the soil.

Because most foundation problems are the result of insufficient density of the underlying soil, grouting is widely used to remediate soil deficiencies under structures that have undergone settlement. The basic principles of application are rather simple and well established.

1. State of the art in grouting technologies for underpinning of old foundations

Permeation grouting for stabilisation of sand is the longest-established and widely used grouting technique. It involves the filling of the pore space in soil. The objective is to fill a void space without displacement of the formation or any change in the void configuration or volume (Warner 2004).

This can be done for strengthening the host formation. Permeation is the only type of grouting that

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can be used in all of the different media into which grout may be pumped.

Grouting of subsoil was first applied over 200 years ago. During the reign of Napoleon in 1802, the French engineer Charles Berigny used a suspension of water and pozzolana cement to fill up caves in the watery gravel ground of a sluice at Dieppe port damaged by settlements to stabilise deposited alluviums. He named it the "procedure for grouting". He made the first sketch of grouting work under a bridge pillar (Kutzner 1996).

In 1810, Berigny used the injection method for reinforcing the supporting base of the bridge over the river Seine in Sèvres, near Paris.

The development of cement grouting continued in France and England throughout the 1800s (Henn 1996). The applications were concentrated on civil structures such as canals, docks, and bridges (Tamaro, Clugh 2001).

The injection of cement grout has been attempted on many occasions. Experience has demonstrated that the method may lead to very satisfactory results, but only provided the soil is relatively homogenous and the grain size is not too small (Xanthakos *et al.* 1994).

The size of cement particles limits the fineness of sand that is suitable for cement grouting. The cement grout will not penetrate the voids of a loose soil with an effective size less than approx. 0.5 mm or a dense soil with an effective size less than about 1.5 mm. Thus, cement grouting is not appropriate for soils much finer than coarse sands (Peck *et al.* 1974).

In 1886, engineer Jeziorsky in Germany patented a water permeable disperse rock reinforcement method used in mining works, when sodium silicate (liquid glass) and subsequently calcium chloride solutions are injected into coarse (rough) or medium coarse sand. Interacting in soil pores as soon as they come in contact with each other, these chemical grout materials on the base of pure solutions end up in silicic acid gel which works on making the sand solid and waterproof. The grouted soil is transformed into impermeable sandstone.

In 1926, engineer of Dutch origin Joosten developed a method proposed by Jeziorsky for civil engineering. Since then, it is globally known as Joosten method (sometimes referred to as Jeziorsky–Joosten method).

Volumes of injected soil greatly increased in 1920s when metro tunnels were intensively built in world's big cities (Paris, London, Berlin and Hamburg); therefore, subsoil of adjacent buildings and sometimes the tunnel arches needed to be reinforced.

After 1930, the Joosten system was applied widely in construction of underground railways. This resulted in increased safety of excavations and decreased risk of structural settlements (Kutzner 1996).

In 1932, in the Soviet Union, engineer Boris Rzhanicyn, collaborating with a well-known Soviet scientists of chemistry of the time, created a soil silicatization by two solutions method, very similar to the one proposed by Joosten. This method was used in Moscow to stabilise sand grounds of buildings and avoid big deformations while building metro tunnels.

In 1939, young Russian scientist Vladimir Sokolovich proposed silicatization by one solution method for fine sands with filtration rate lower than 2 m/day, in which case silicatization by two solutions cannot be used.

The use of non-organic and organic multimolecular compound based solutions started along with cement suspension, as the latter was suitable for injection into gravel only and could not penetrate into soils with smaller particles.

Chemical grout materials of very low viscosity were developed to be readily mixed on the surface and injected into the subsoil in one shot. Such materials allow stabilising soil down to fine sand with a small content of silt (Krizek, Spino 2000).

French engineers Lemaire and Dumaunt patented a method that requires mixing sodium silicate with acid and injecting this mixture into fine sand, where it turns into weak waterproof gel. This method was further developed by Geyrard, Rodio and others.

New ways of ground stabilisation by injection were used along with Joosten method in economically developed countries in 1930s: Francois method (using sodium silicate and aluminium sulphate mixture), Rodio's (liquid glass with limewater) and other.

Sodium silicate based solutions are the most commonly used non-organic chemical substances for soil injection, and some synthetic resins are successfully used for grouting.

Many other chemical processes based on the one-shot principle were developed aiming to obtain a very low viscosity at the time of injection with only a slow increase in viscosity until gelation occurs, thus ensuring maximum penetration (Gouvenot 1996).

Polymeric resins allow reinforcing sand of various grain size distribution and mineral consistence (even the fine-grain sand with filtration rate of 1 m/day), making it stable and waterproof.

Multimolecular organic solutions are successfully used not only for underpinning foundations of ancient buildings (Littlejohn 1985; Brunner 2003; Aslay 2007), but also for bridge ground reinforcement, tunnel stabilisation (Falk, Burke 2003), and cracked rock reinforcement (Cermak, Fennimore 2001).

Generally, grout can be used if permeability of the deposit is greater than 0.001 cm/s. One of the principal precautions with grouting is that the injection pressure should not be sufficient to lift the ground surface and to damage adjacent buildings (Sheen 2002).

Geotechnical grouting needs verification and has a test program to verify grouting effectiveness (Atwood, Lambrechts 1995).

Penetration grouting is used for scour remediation (Fahoum, Baker 1998).

In the wake of the World War II, when the use of synthetic resins for grouting started, the improved physical and mechanical soil characteristics (waterproof, high compression strength, durability) were greatly applauded and for at least some time, there were no concerns regarding the impact of polymeric solutions on the environment.

Today, it cannot be withheld that polymeric resins used for ground reinforcement in 1940s–1970s had toxic components that could have had negative effect on the environment and human health.

The issue was raised in Tokyo in 1977, at the 19th conference of International Society for Soil Mechanics and Geotechnical Engineering (ISSMGE), where Japanese researchers Ando and Makita talked about a family that drank water from a well which stood next to a construction site where soils were stabilised by acrylic *Sumisoil* resin. All family members suffered from health disorders and were hospitalised (Ando, Makita 1977).

Environmental concerns resulted in rejection of some polymeric resins and modification of other resins (carbamide, acrylic) in order to make them environmentally acceptable.

Deep soil grouting is useful for safe reconstruction of old communications and services in big cities (Ulitsky, Bogov 2000).

Under favourable ground conditions, grouting is a useful underpinning method used in case of deep excavations undertaken close to existing structures (Thorburn, Hutchison 1985). Injections are made from ground level, thus obviating the need for shoring or needling, while the wall of consolidated ground acts as a retaining wall during excavations close to existing foundations.

The new steps of grouting technologies are bio-grouting using urea and calcium chloride (Suer *et al.* 2009), permeation grouting using organosilane (Daniels, Hourani 2009), and limited mobility grouting (Warner, Byle 2012).

From the early days of simple slurry injection to current sophisticated techniques, grouting has played, and continues to play, an important role in remediation and new construction worldwide. As new grouting technology is developed, and existing technologies are refined, the range of applications increases (Welsh, Burke 2000).

2. Grouting with multimolecular organic solutions

Synthetic resins are solutions of multimolecular organic oligomers with a relatively small molecular mass, and when hardening, they turn into insoluble and infusible polymers with dimensional structures.

Out of many synthetic resins for soil stabilisation, carbamide, acrylic, urethane, furan and resorcinol–formaldehyde resins proved to be the most suitable.

Carbamide resins form when carbamide (urea) are polycondensed with formaldehyde. They demonstrate thermoreactive characteristics: in higher temperature and under some conditions, in normal temperature, from a flowing state, they turn into viscous-flowing state, and later – into a hard, insoluble and irreversible state.

In 1929, the American chemist Carothers offered grouping polymers into polycondensed and polymerized. According to Carothers, polycondensed polymers are formed from polyfunctional monomers, relieving products with small molecular mass (e.g. water). Polymerized polymers form from monomers but do not relieve any other products. Carbamide resin is a polycondensed polymer.

The American engineer Lawton was the first to investigate the carbamide resin for its use in soil stabilisation. In 1947, he used the resin to reinforce watery sand in an oil well. He used ammonium chloride as a resin hardener.

Later, carbamide resin was used to stabilise soils in many different countries. It reliably reinforced fine-

grain sand and coarser sand soils, provided high compression strength (of up to 10 MPa), water resistance and durability. Solutions of hydrochloric acid (3 or 5 per cent), organic oxalic acid (6%) and ferric chloride (18%) were used as hardeners.

Owing to their availability and low cost, urea–formaldehyde resins have come into widespread use in world geotechnical engineering for soil stabilisation.

Acrylic resins are the polymers and copolymers of acrylic and metacrylic acids, nitriles and ethers. Americans Houser and Dannenberg were the first to announce a possibility to use acrylic resins for soil reinforcement. Since 1950, geotechnical engineers have been successfully using acrylamide formulas in ground reinforcement. The most popular of these are American AM-9, PWG, Q-Seal, AC-400, AV-100 and Japanese *Sumisoil*.

Acrylamide formulas allowed getting a weak solution, however, a waterproof gel (solution that hardens during the gelatinization process). From environmental perspective, some of them were not clean. For instance, the effect of acrylamide on the Japanese family that used water from a well only 5 m away from a construction site where soils were being reinforced by AM-9 solution is described in literature of geotechnical engineering. The free acrylamide monomer penetrated into the well and resulted in a 1 g/m³ concentration. That was enough to cause neural and mental disorders to the family members. All family members successfully recovered after they had been hospitalised (Ando, Makita 1977).

Following this and some other incidents, the National Institute for Occupational Safety and Health in the USA has forbidden using the catalysts of AM-9 formula beta-dimethylaminopropionitrile (β-DMAPN).

Modern acrylic resins meet today's hygienic and sanitary requirements; thus, they can be used for ground reinforcement in construction.

Other synthetic resins were used more rarely for ground stabilisation and foundation underpinning.

3. Laboratory testing of carbamide resin solutions determined to grout sandy soil

The author of this paper investigated the following properties of carbamide resin solutions: density, viscosity, pH (alkalinity level), and evaporation of components from solution.

Table 1. Relation between composition and density of solution

Composition of solution			Mark	Density, g/cm³
Resin KM-2	Water	Urea		
100	0	0	–	1.162
100	50	0	"0"	1.110
100	50	5	"5"	1.115
100	50	10	"10"	1.121
100	50	15	"15"	1.126
100	50	20	"20"	1.132
100	50	25	"25"	1.137
100	50	30	"30"	1.142

Density of carbamide resin solution increased with increase of urea addition mass (Table 1). Density is measured with standard densimeter with the precision of 0.001 g/cm³.

The viscosity of a fluid is a measure of its resistance to gradual deformation by shear stress or tensile stress. For liquids, it corresponds to the informal notion of "thickness". Relation between composition and viscosity of solution is given in Table 2.

Table 2. Relation between composition and viscosity of solution

Mark	Time of effusion, s	Proportional viscosity, cP
"0"	63.0	10.71
"5"	53.6	9.15
"10"	49.8	8.55
"15"	46.8	8.07
"20"	45.2	7.83
"25"	43.8	7.63
"30"	43.2	7.55

In chemistry, pH (alkalinity level) is a measure of the acidity or basicity of an aqueous solution. Solutions with a pH less than 7 are acidic and solutions with a pH greater than 7 are basic or alkaline. Pure water has a pH very close to 7.

Alkalinity level pH increased linearly with the increase of additive – crystalline urea – in the resin solution (Fig. 1).

The content of free formaldehyde in carbamide resins used for injection chemical stabilisation of soils attains 2%. Its release increases sharply in the hardening process of the resins; however, a significant portion
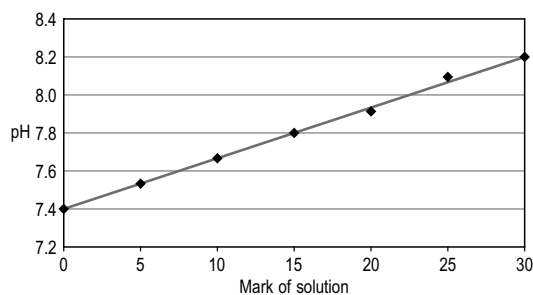
Fig. 1. Relation between mark and pH of solution

Fig. 2. Dependence of free formaldehyde evaporation from resin solutions to air

of the water contained in the resin, and released during its condensation, goes over into the bonded state where it cannot absorb and retain free formaldehyde that is released. Therefore, the latter passes from the stabilised sandy soil to the environment; this is also promoted by the low boiling point of formaldehyde (−19.2 °C).

It is known that free formaldehyde dissolves readily in water, forming a saturated formalin solution with a formaldehyde content of up to 37% at normal temperature, and mixes readily with air in any proportion. The density of gaseous formaldehyde is close to that of air; this permits the free circulation of formaldehyde that is released into the atmosphere.

The maximum allowable concentration (MAC) of formaldehyde in the air should not exceed 0.5 mg/m^3 for industrial and 0.012 mg/m^3 for residential air space. The MAC should not be higher than 0.005 mg/litre in agricultural, drinking water and recreational water supply, and 0.1 mg/litre for fish breeding.

Experience has shown, that the content of free formaldehyde in air and water media in the vicinity of segments of soil stabilised with carbamide resins significantly exceeds these values. An especially large amount of formaldehyde is released into the atmosphere during the opening of mining excavations in soils or rock stabilised with carbamide resin.

The amount of free formaldehyde that passes from resin solution and from stabilised soil into the air and water media was determined using the titrimetric method developed by the author, which is based on the interaction of formaldehyde with a neutral solution of sodium sulfite, as a result of which a formaldehyde-bisulfite addition compound is formed with the release of an equivalent amount of free sodium hydroxide. The latter is titrated with hydrochloric acid, the amount of which is a measure of the content of free formaldehyde in the sample under investigation. Dependence of free formaldehyde evaporation from resin solutions to air is given in Figure 2.
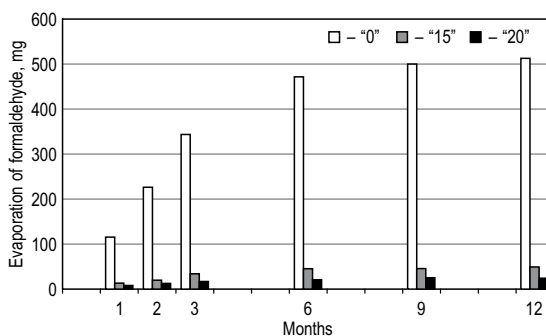
To determine free formaldehyde that passes from the stabilised soil into an aqueous medium, a sample is placed in a dry jar which is tightly sealed with a polyethylene cover to avoid loss of free formaldehyde. Specimens taken from the jar are rapidly crushed into pieces ranging from 3 to 10 mm in size to form a batch weighing 50–60 g. It is then placed in a half-litre glass jar, and covered with 100 ml of distilled water, after which the jar is tightly sealed with a polyethylene cover. 24 hours after, the water is filtered from the jar through a paper filter into a 250 ml conical flask. The soil sample is wetted with a small amount of distilled water, which is added to the filtrate via the filter.

The specimen under investigation is again allowed to sit in water for 24 h. After the indicated time, the water is changed with subsequent determination of free formaldehyde in it.

The test is discontinued in time with the complete absence of free formaldehyde in the aqueous medium. Several drops of phenolphthalein and 0.5 normal (N) solution of sodium hydroxide are added to the filtrate in the conical flask to the appearance of a week crimson-colour solution.

Yet another drop of phenolphthalein and 25 ml of a saturated sodium sulfite solution neutralised in accordance with the phenolphthalein by the several drops of 0.5 N HCl are added to the prepared solution. The contents of the flask are agitated and allowed to rest for 5 min, after which the alkali that has been released is titrated with a 1 N solution of HCl until the solution loses its crimson colour.

A "control" test is conducted concurrently to improve the accuracy of the determination; 100 ml of distilled water and the above-indicated volume of neutralised sodium sulfite solution is taken for this test, but without introducing the batch of stabilised

Table 3. Release of free formaldehyde from stabilised soil in air medium

Time-span of investigation	Relsease of free CH$_2$O in percent to mass of resin by different compositions				
	"0"	"5"	"10"	"15"	"20"
1 day	0.05	0.02	0.01	0.004	0.002
4 days	0.19	0.07	0.04	0.01	0.01
7 days	0.29	0.13	0.06	0.03	0.01
10 days	0.40	0.18	0.09	0.04	0.02
14 days	0.55	0.24	0.12	0.05	0.02
21 day	0.75	0.33	0.16	0.07	0.03
28 days	0.92	0.40	0.20	0.08	0.04
2 months	1.31	0.57	0.28	0.12	0.05
3 months	1.63	0.71	0.35	0.15	0.07
6 months	1.97	0.86	0.42	0.18	0.08
9 months	2.11	0.93	0.45	0.19	0.09
12 months	2.19	0.96	0.46	0.20	0.09

soil. Release of free formaldehyde from stabilised soil in air medium is given in Table 3.

The amount of free formaldehyde passing from the stabilised soil into the air is determined in a manner similar to the above-described method, only the crushed soil is placed in a polyethylene or glass cup, which is mounted in a half-litre glass jar above water surface. As in the previous case, the jar with the specimen is tightly sealed with a cover. The free formaldehyde from the specimen will first pass into the air medium, and then be absorbed by the water, which is subject to determination. In contrast to the previous determination, the holding time of the specimen above the water is increased by a factor of two.

Under laboratory conditions, by the experimental impregnation of a certain mass of sand with carbamide resin, and under field conditions from the average consumption of resin corresponding to 380 litres with a density of 1.10 g/cm^3 per 1 m^3 of stabilised sand is established.

It is apparent from Table 3 that 10–13 and 21–25 times less formaldehyde is released into the air from soil stabilised with the modified resins "15" and "20" than from the soil stabilised with the unmodified resins, respectively. In this case, the sharp irritating odour of formaldehyde was perceived above the freshly crushed specimens of soil stabilised with the unmodi-

fied resin, and acute smarting of the eyes was sensed, while these phenomena were absent in specimens of the soils stabilised with the modified resins.

As the investigations indicated, free formaldehyde contained in soils stabilised with the resin is almost completely released into the aqueous medium during the course of several days in the case where the soils are fine-crushed. Its release into the aqueous or air medium will be determined by the specific surface of the soil, which is a function of the reduction factor of the crumbled soil.

Conclusions

1. Multimolecular organic solutions on base of carbamide resin are acceptable for sandy deposits grouting by underpinning of old foundations.
2. Addition of active components to carbamide resins increase density and pH (alkalinity level) of solution, decrease viscosity and evaporation of components from solution.
3. 10 to 25 times less formaldehyde is released into the air from soil stabilised with the modified resins than from the soil stabilised with the unmodified resins.

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SĄVEIKA TARP SMĖLIO NUOSĖDŲ IR DAUGIAMOLEKULIŲ ORGANINIŲ TIRPALŲ STABILIZUOJANT GRUNTĄ

R. Mackevičius

Santrauka. Straipsnyje pateikta injekcijos technologijų taikymo senų pamatų pagrindui stiprinti apžvalga, injekcijų daugiamolekuliais organiniais tirpalais analizė ir dispersinių gruntų, stabilizuotų organiniais polimerais, vienerių metų laikotarpio tyrimų rezultatai. Buvo palygintos nemodifikuotų ir modifikuotų karbamidinių dervų savybės, tiriamos tokios dervos tirpalų savybės, kaip tankis, klampumas, pH (vandenilio jonų rodiklis) ir komponentų išgaravimas iš tirpalo. Kitame tyrimų etape lyginamos aliuvinio vidutiniškai tankaus smėlio, stabilizuoto nemodifikuotomis ir modifikuotomis dervomis, savybės.

Reikšminiai žodžiai: grunto stabilizavimas, organiniai tirpalai, smėlis, cementacija, pamatai, injekcija, pagrindo stiprinimas, ekologinė sauga.

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