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Low Viscosity Pore Fluid to Manufacture Transparent Soil

ABSTRACT: Transparent soil has been investigated for its potential as a substitute research media for natural soil. The mechanism for manufacturing the transparent soil is through adding an appropriate pore fluid to silica gel particles with the same refractive index. Two types of high viscosity pore fluids were identified by Iskander in 1994. However, because of the high viscosity of these two pore fluids, air was easily entrapped, which made the manufacture of a large mass of transparent soil difficult. In addition, the identified pore fluids caused serious membrane deterioration during triaxial laboratory testing. This research presented herein is an experimental investigation on low viscosity pore fluids to manufacture transparent soil, including the fluid/particle interaction in the stimulant matrix. Two low viscosity pore fluids were identified with minimum interaction with latex membranes.

KEYWORDS: low viscosity pore fluids, transparent soil, silica gel, refractive index

Introduction

Transparent soil has been used in the soil laboratories to “see” and study the deformation behavior of soils in the model test under different loading conditions (Sadek et al. 2003; Toiya et al. 2007; Song and Hu 2009). It has also been used to study the flow pattern and contaminant transport in porous media (Welker et al. 1999). Transparent soil is a mixture of pore fluid and granular silica gel or amorphous silica gel. It becomes transparent because the pore fluid has the same refractive index of 1.448 as silica gel at 25 °C. Granular silica gel has either a granular or beaded shape. It is made by partially dehydrating metasilic acid and is commonly used as a moisture absorbent, a catalyst, or a purifier of different substances. Silica gels and powders are able to absorb pore fluids and thus drive air out of the internal pores (Mannheimer and Oswald 1993). Therefore, an appropriate pore fluid is critical to manufacture transparent soil. Iskander et al. (1994) discovered two matching pore fluids. The first pore fluid is a 50:50 blend by weight of a colorless mineral (Drakeol 35) and normal paraffinic solvent (Norpar 12). Its refractive index is 1.447 at 24 °C, and its viscosity is 5.0 cP at 24 °C. The density of the pore fluid is 0.804 g/cm³. The second matching pore fluid is a mixture of calcium bromide and water. The refractive index and viscosity of the mixture are 1.448 and 3.6 cP, respectively, at 25 °C. Depending on the pore fluid used to manufacture transparent soil, the dry unit weight of silica gel is 6–9 kN/m³, and its saturated unit weight is 11–14 kN/m³. Welker et al. (1999) adopted transparent soil to investigate the flow patterns in prefabricated vertical drains. They found difficulties using the transparent soil mentioned above, such as incompatibili-

ties with the latex membranes, high viscosity of the pore fluid, degradation in the transparency for the large samples, special scaling requirements for the experiment, and high costs. The geotechnical properties of transparent silica have been studied by Sadek et al. (2002) and Iskander et al. (2002a, 2002b).

To correctly and properly simulate the behavior of natural soil, the selected pore fluid needs to have the following properties: (1) Kinematic viscosity identical or close to that of water; (2) surface tension close to that of water; (3) incompressibility; (4) affordability; and (5) low and no interaction with silica gel, latex membranes, and the model container. In this paper, 12 types of chemical fluids were selected and tested in order to identify and determine the appropriate mixture of pore fluids to manufacture the transparent soil. Viscosity and refractive index tests were carried out on the selected fluids. The interaction of the candidate matching pore fluid with the latex membrane was also investigated.

Testing Program

Temperature Variation of the Viscosity and Refractive Index of the Potential Solvents

Twelve (12) different organic fluids were first selected as the possible fluid. These fluids were selected based on their refractive index and their viscosity. Low viscosity pore fluids with refractive index greater than 1.448 and less than 1.448 were selected. Those with refractive index greater than 1.448 were taken as base pore fluid; those with refractive index less than 1.448 were taken as the compensating pore fluid.

To identify the relationship between the refractive index and temperature, an AR200 Digital Handheld Refractometer manufactured by Reichert Analytical Instruments, Depew, NY, was used. It can measure a wide range of refractive indexes from 1.3300 to 1.5600 to an accuracy of ± 0.0001 nD. All solvents were put into a temperature chamber, and their refractive indexes were measured at six different temperatures (10, 15, 20, 25, 30, and 35 °C), which cover the possible range of operating temperatures.

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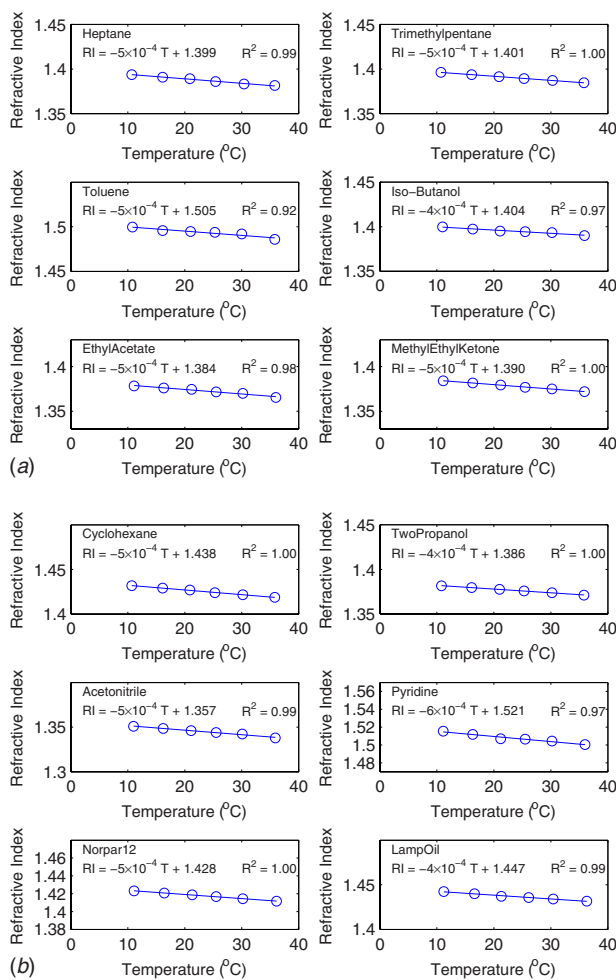


FIG. 1—[(a) and (b)] The refractive index variation of the chemical solvents with the temperature.

The viscosity of the chemical solvents was also measured by Cannon–Fenske viscometer tubes at six different temperatures (10, 15, 20, 25, 30, and 35 °C) to examine their variation against temperature. Two different sizes of tubes were used. Size No. 25 was suitable for the viscosity ranging from 0.5 to 2 cSt, while size No. 75 was suitable for the viscosity ranging from 1.6 to 8 cSt. All the tests were carried out following ASTM D445-09 (2009). The variation of refractive index with temperature for the tested pore fluids is shown in Fig. 1. The refractive index of all tested pore fluids showed a linear variation with respect to temperature within the tested temperature range but with different slopes. The variation of viscosity with temperature for the tested pore fluid is shown in Fig. 2. The viscosity of all the tested pore fluids also showed a linear variation with respect to temperature within the tested temperature range (Mallinckrodt Baker, Inc. 2009).

Determination of the Matching Refractive Index of the Matching Pore Fluid

The dry silica gel has a refractive index of 1.448 at 25 °C. When the pore fluid has the same refractive index, the silica gel will become transparent. Among those tested candidate pore fluids, those with a refractive index greater than 1.448 were selected as the base pore fluid. Those with a refractive index less than 1.448 were selected as

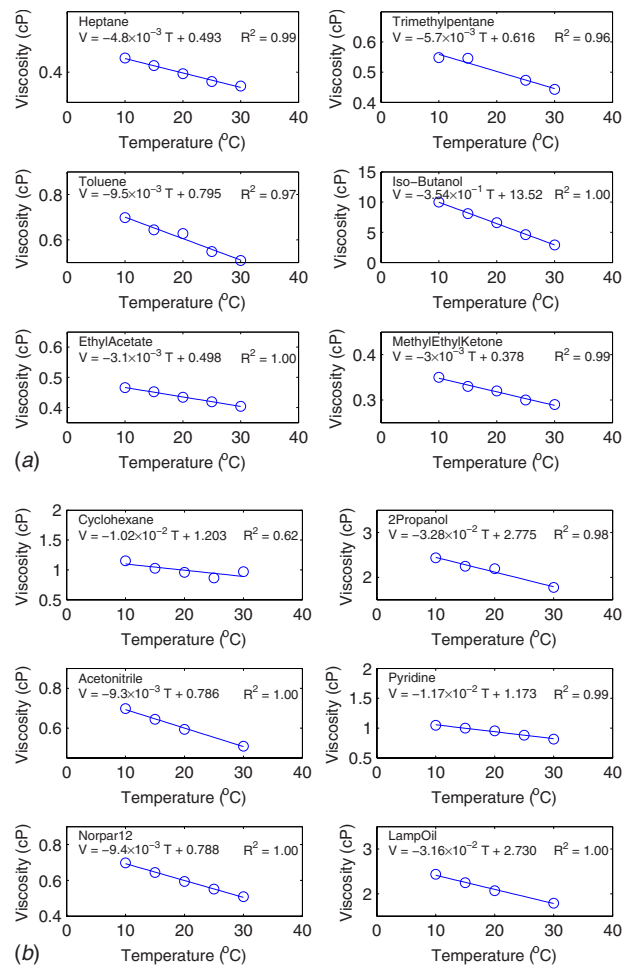


FIG. 2—[(a) and (b)] The viscosity variation of the chemical solvents with the temperature.

the compensating pore fluid. In order to obtain a pore fluid with the matching refractive index to get the best transparency effects (can be clearly seen through) for the silica gel, 4 mL of the base pore fluid with a refractive index greater than 1.448 (toluene, lamp oil, or pyridine) was first put into the glass tube (diameter of 1.50 cm), and 3–4 g of dry silica gel was added into the tube. Then, the compensating pore fluid (acetonitrile, cyclohexane, ethyl acetate, *n*-heptane, iso-butanol, methyl ethyl ketone, 2 propanol, 2,2,4-trimethylpentane, and Norpar 12) with a refractive index less than 1.448 was added gradually until the best transparency effect was achieved. The volume ratio between the base pore fluid and compensating pore fluid was then recorded, and its refractive index was measured. During the test, the temperature was kept at 25 °C. Toluene was found to be a good base pore fluid, which a target refractive index can be obtained by mixing with 2 propanol, acetonitrile, ethyl acetate, methyl ethyl ketone, and iso-butanol. However, pyridine cannot make the dry silica gel become transparent when mixed with any other solvent. The pore fluid obtained from pyridine and another pore fluid could not get into the pores of the silica gel. When the compensating pore fluid was added into the base pore fluid with dry silica gel inside, the interaction between the pore fluid and the silica gel is heat released, and the air bubbles inside the pores are coming out of the pore fluid. The amount of heat released can be felt through the temperature of the testing glass tube. When cyclohexane was added to the silica gel with toluene, air bubbles

TABLE 1—Test results of trial mixing to obtain the matching refractive index pore fluid.

Mixing Fluid	Transparency Effect	Volume Ratio	Refractive Index (25°C)	Interactions Observed
Toluene+cyclohexane	Poor	NA	NA	A moderate amount heat released
Toluene+2 propanol	Good	1.07:1	1.448	None
Toluene+acetonitrile	Good	1.08:1	1.448	A large amount heat released
Toluene+2,2,4-trimethylpentane	Poor	NA	NA	None
Toluene+ethyl acetate	Good	0.72:1	1.448	A large amount of heat released
Toluene+methyl ethyl ketone	Good	0.95:1	1.448	A large amount heat released
Toluene+heptane	Poor	NA	NA	Turned into yellow color
Toluene+iso-butanol	Good	2.22:1	1.448	None
Toluene+Norpar 12	Poor	NA	NA	None

Note: NA=not available.

were observed coming out of the pores of the silica gel but not as quick as when ethyl acetate was added to the silica gel with toluene; no obvious heat was released. The behavior observed when adding the compensating pore fluid to the base solvent toluene was described in Table 1. When methyl ethyl ketone was added into the base pore fluid of toluene, a large amount of heat was released; the temperature rise of the tube was felt by the hand. There is an interaction happened between the silica gel and the pore fluid. This does not influence the transparency of the silica gel and the pore fluid. Further investigations need to be performed on the geotechnical properties of the transparent soil made with different types of pore fluid.

The second base fluid investigated in this research was lamp oil, which is a liquid petroleum product designed to burn cleanly in brass and glass oil lamps, torches, and lanterns. Lamp oil is from the same family such as kerosine and has been further processed and refined to reduce the harmful smoke, soot, and other pollutants as kerosine. The lamp oil investigated in this research is 99 % pure liquid wax paraffin produced by Lamplight Farms, a Division of the W.C. Bradley Co. It was available at a local supermarket. The test results showed that lamp oil can be mixed with 2 propanol, acetonitrile, 2,2,4-trimethylpentane, ethyl acetate, iso-butanol, and cyclohexane to reach the refractive index of dry silica gel. The test results are summarized in Table 2. When the dry silica gel was added to the mixture of lamp oil and ethyl acetate, air inside the silica gel pores quickly came out, and a large amount of heat was released. It took about 1 h to make the silica gel become transparent. However, the transparency deteriorated when used to make a 6 cm thick transparent soil mass.

Investigation on the Interaction between the Pore Fluid and the Latex Membrane

The low viscosity pore fluids tested in this research are organic chemical solvents. The degradation of a latex membrane when ex-

posed to these chemical solvents has been a concern during triaxial tests. Iskander et al. (2002a, 2002b) observed the deterioration of the latex membrane when using a pore fluid of Drakeol 35 and Norpar 12. To investigate the interaction between a pore fluid and the latex membrane, a series of immersion tests was conducted following ASTM D543-06 (2006). It includes two measurements of changes in (1) weight and dimension and (2) mechanical properties of the latex membrane.

Immersion Test—The latex membrane from Humboldt Mfg. Co. was selected as the testing material since it is commonly available at the laboratory for soil testing. The membrane was cut into a specimen size of $2.54 \times 7.62 \times 0.0636 \text{ cm}^3$ ($1 \times 3 \times 0.025 \text{ in.}^3$) and weighed before immersion. Then, the test specimen was completely immersed into the pore fluid for 7 days, allowing the total surface area to be exposed to the fluid. After 7 days, the specimen was removed from the container and wiped dry; then its weight and dimensions were measured. Observations were made on the appearance of the surface for each specimen after exposure to the pore fluid. The membranes were air dried for 40 h, and then they were weighed and measured to examine any changes. Figure 3 shows the membranes after 40 h of air drying and the following observations were made. (1) The pore fluid of toluene and 2 propanol mixture did not change the color of the membrane, which is a sign of low interaction. (2) A moderate deterioration in color of the membrane immersed into the mixed fluid of toluene and the methyl ethyl ketone, mixed fluid of toluene and acetonitrile, mixed fluid of toluene and iso-butanol, and the mixed fluid of lamp oil and 2 propanol was observed. The membrane immersed into the mixed fluid of toluene and ethyl acetate, lamp oil and methyl ethyl ketone, and lamp oil and ethyl acetate had the strongest color deterioration. (3) The membrane immersed into the pore fluid of lamp oil and 2 propanol, toluene and iso-butanol, lamp oil and methyl ethyl ketone, and lamp oil and ethyl acetate curled. The immersion test data are shown in Table 3. The data show that after the immersion, the

TABLE 2—Mixture of lamp oil and the compensating solvents with dry silica gel.

Mixing Fluid	Transparency Effect	Volume Ratio	Refractive Index (25°C)	Interactions Observed
Lamp oil+cyclohexane	Poor	NA	NA	None
Lamp oil+2 propanol	Good	1:0.15	1.434	Heat released
Lamp oil+acetonitrile	Poor	NA	NA	Separation found
Lamp oil+2,2,4-trimethylpentane	Poor	NA	NA	None
Lamp oil+ethyl acetate	Good	1.28:1	1.434	Heat released; transparency deteriorates with large sample
Lamp oil+methyl ethyl ketone	Good	5:1	1.434	None. Transparency deteriorates with large sample
Lamp oil+heptane	Poor	NA	NA	None
Lamp oil+iso-butanol	Poor	NA	NA	Heat released
Lamp oil+Norpar 12	Poor	NA	NA	None

Note: NA=not available.

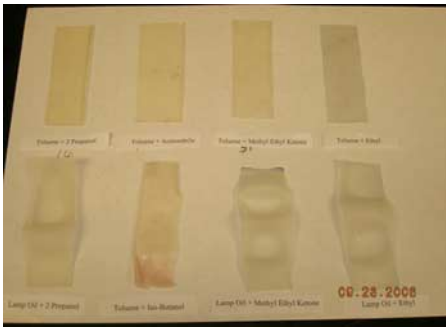


FIG. 3—The latex membrane 40 h after the immersion test.

weight of the membrane greatly increased due to the absorption of the pore fluid. There was also an increase in the length, width, and thickness for the membrane immersed in all the solvents. Meanwhile, there was an increase in the density for the membrane immersed in the pore fluid of toluene and 2 propanol, toluene and acetronile, toluene and ethyl acetate, toluene and methyl ethyl ketone, and toluene and iso-butanol. However, the density for the membrane immersed in the pore fluid of lamp oil and 2 propanol, lamp oil and ethyl acetate, and lamp oil and methyl ethyl ketone decreased.

Tensile Test—The tensile test was performed on the latex membrane immediately after the immersion test. A low load capacity of 0.045 kN (10 lb) and high sensitivity load cell was used in performing the tensile test because the ultimate tensile strength of the latex membrane was very low. The deflection rate was taken as 0.085 cm/s (2 in./min). The original size of the specimen for the tensile test before the immersion was 3.81 cm (1.5 in.) in width and 76.2 cm (2.5 ft) in length. After the 7-day's immersion test, the specimen was taken out and wiped dry. Then, it was cut into 12.7 cm (5 in.) pieces for the tensile test. The tensile test for the membrane was set up as shown in Fig. 4. A piece of sand paper was put between the grip and the membrane to make sure that the grip can hold the tested membrane firmly. The membrane could be stretched to six times of its original length. The detailed information for the tested latex membrane specimens is listed in Table 4. The thickness and width of the specimen were measured after the immersion test before the tensile test. The length of the tested specimen was measured after the specimen set up on the testing machines. Because of the different grip length of each specimen, the tested specimen length is different as listed in Table 4. During the testing process,



FIG. 4—The experimental set up for the tensile test of the latex membrane.

sample latex 28 slipped from the grip because the sample became very thin when stretched. Latex 14 was kinked during the test. These conditions do not affect the testing results since the membranes have been stretched to six times of the original size, beyond the range of interests. The stress-strain curve for the tested membranes is plotted in Fig. 5. Latex 0 was the original Humboldt membrane before any treatment. The test results showed that there was a strong degradation for the latex membranes immersed in the chemical solvents of the mixture of toluene and acetronile (latex 10), lamp oil and 2 propanol (latex 54), lamp oil and methyl ethyl ketone (latex 13), and lamp oil and ethyl acetate (latex 17) in both the tensile strength and elasticity. For the latex membrane immersed in the water (latex 1), the membrane's initial elastic modulus increased. However, the membrane lost most of its elasticity, being easily torn apart. The latex membranes immersed in the mixture of toluene and acetronile (latex 10), lamp oil and 2 propanol (latex 54), lamp oil and methyl ethyl ketone (latex 13), and lamp oil and ethyl acetate (latex 17) were broken at a low stress and also at a small strain level. The latex membranes immersed in the mixture of toluene and methyl ethyl ketone (latex 14) and toluene and 2 propanol (latex 28) only had a slight degradation. For the membranes immersed in the mixture of toluene and ethyl acetate (latex 37), there was a drop in the tensile strength of the membrane, but the elasticity of the membrane remained constant. Therefore, it was concluded that the mixtures of toluene and methyl ethyl ketone, and toluene and 2 propanol would be the best choices for the pore fluid. The mixture of toluene and ethyl acetate was also recommended to

TABLE 3—7-day immersion testing data on Humboldt membranes.

Testing Pore Fluid	Time	T+2 pro	T+Ace	T+EA	T+MK	T+IB	LO+2 pro	LO+EA	LO+MK
Mass change (%)	7days	+185.58	+53.17	+260.92	+220.95	+245.28	+262.96	+278.18	+282.76
Length change (%)	7 days	+35.06	+9.74	+46.75	+38.16	+41.56	+53.85	+57.69	+61.04
Width change (%)	7 days	+32.81	+20.69	+48	+38.46	+48.44	+60.00	+60.78	+67.33
Thickness change (%)	7 days	+53.85	0	+46.15	+46.15	+7.69	+53.85	+53.85	+57.69
Density change (%)	7 days	+3.48	+15.65	+13.70	+14.79	+52.58	-4.16	-3.05	-7.67
Mass change (%)	40 h	+10.26	+5.31	+7.35	+8.19	-1.89	+31.48	+32.73	+43.10
Length change (%)	40 h	0	-1.30	-1.30	0	+1.82	+6.41	+15.38	+12.99
Width change (%)	40 h	+1.56	+4.83	+4.00	0	-1.95	+8.00	+1.96	+5.58
Thickness change (%)	40 h	+7.69	-7.69	0	0	-15.38	0	0	+10.77
Density change (%)	40 h	+0.81	+10.26	+4.58	+8.19	+16.15	+14.41	+12.82	+8.30

Note: T+2 pro=toluene+2 propanol; T+ace=toluene+acetonitrile; T+EA=toluene+ethyl Acetate; T+MEK=toluene+methyl ethyl ketone; T+IB=toluene+iso-butanol; LO+2 pro=lamp oil+2 propanol; LO+EA=lamp oil+ethyl acetate; and LO+MEK=lamp oil+methyl ethyl ketone.

TABLE 4—The tested latex membrane size and testing observation for the tensile test.

Sample Description	Sample Number	Thickness (mm)	Width (mm)	Length (mm)	Testing Description
L+2 propanol	Latex 54	0.457	58.0	97.1	Membrane being torn apart
L+MEK	Latex 13	0.508	64.4	103.0	Slipped from grip, nearly exceeded the testing device capacity Used sand paper to suppress slipping, nearly exceeded the extension limit, aborted
T+ace	Latex 10	0.356	39.0	65.1	
T+ethyl acetate	Latex 37	0.356	38.4	78.7	Sand paper used
T+2 propanol	Latex 28	0.330	40.6	64.1	Membrane slipped from the grip
L+ethyl acetate	Latex 17	0.305	59.6	68.1	Membrane being torn apart, sand paper used
Water	Latex 1	0.381	38.1	70.1	Sand paper used, membrane slipped from the grip
T+MEK	Latex 14	0.381	39.5	56.3	Sand paper used, membrane kinked, load dropped
Original membrane	Latex 0	0.381	38.6	56.6	Sand paper used, membrane kinked

Note: T+2 propanol=toluene+2 propanol; L+MEK=lamp oil+methyl ethyl ketone; T+ace=toluene+acetonitrile; T+ethyl acetate=toluene+ethyl acetate; and T+MEK=toluene+methyl ethyl ketone.

manufacture transparent soil. The lamp oil was not recommended to make the transparent soil since it greatly reduced the tensile strength and elasticity of the latex membrane (latex 13, latex 17, and latex 54), as shown in Fig. 5.

Summary and Conclusions

This research program investigated twelve (12) different types of chemical solvents to identify low viscosity pore fluid to manufacture transparent soil together with the silica gel. Eight potential matching pore fluids were further investigated to examine their interactions with the latex membrane. The test results show that the mixtures of toluene and methyl ethyl ketone, toluene and 2 propanol are the two best low viscosity pore fluid with their very low interactions with the latex membrane. These two types of low viscosity pore fluids are harmful to the human body, and the protective measures (wearing safety masks and plastic gloves and operating in a venting hood) must be taken when using them to manufacture the transparent soils. Further research on the identification of a pore fluid of low viscosity, which is nonhazardous and does not interact with the silica gel or the plastic membrane, is greatly encouraged. It is noted in other studies which triaxial tests have been successfully conducted with Nitril rubber membranes (Iskander et al. 2002a). Double latex membranes have also been used so that the deteriorated membrane is supported by another intact membrane.

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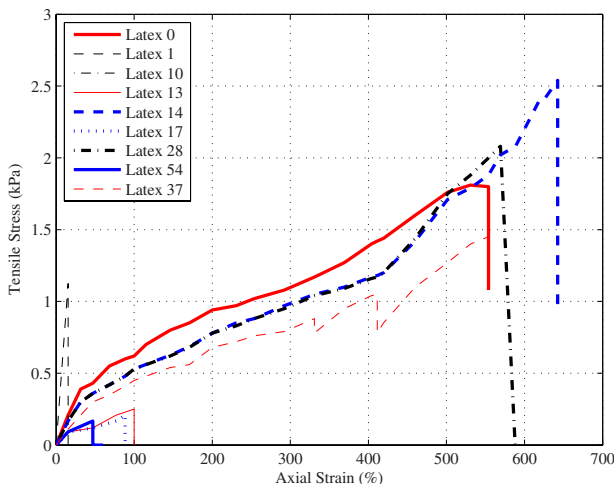


FIG. 5—The stress-strain curve for the tested membrane.

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