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EFFECTS OF LIQUID POLYMERS ON THE STRENGTH AND FREEZING-THAWING PROPERTIES OF BENTONITE AND KAOLIN CLAYS: A COMPARATIVE STUDY FOR COLD CLIMATES

ZEYNEP NESE KURT ALBAYRAK*, MEHMET AKIF ISIK

Department of Civil Engineering, Faculty of Engineering, Ataturk University, Erzurum, Turkey

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Abstract. Clay soils can exhibit swelling and settlement behaviour when interacted with water. The clays located in road infrastructure can damage the road pavement and cause cracks because of swelling and/or settlement of clays. The freezing-thawing processes of clay soils can increase the damages of pavement. Additionally, the strength losses can occur. Weak soils can be improved using various soil improvement techniques with or without additives. In this study, liquid polymers (polyurethane, epoxy resin, and styrene acrylic) were combined with clays in various amounts (5 wt.%, 10 wt.%, 15 wt.%, and 20 wt.%) to improve the strength and investigate the freezing-thawing behaviour of high-plasticity bentonite and low-plasticity kaolin clay. Unconfined compression tests and freezing-thawing cycles were performed on the samples with additives that were cured for 1 and 7 days. As a result, it was observed that polyurethane, epoxy resin, and styrene acrylic additives had positive effects on both the unconfined compressive and the post freezing-thawing unconfined compressive strengths of low and high plasticity clays in cold climates.

* Corresponding author. E-mail: znkurt@atauni.edu.tr

Zeynep Nese KURT ALBAYRAK (ORCID ID 0000-0002-6323-8652)
Mehmet Akif ISIK (ORCID ID 0000-0001-8646-5308)

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Keywords: bentonite, freezing-thawing, kaolin clay, liquid polymer, stabilization, strength.

Introduction

In civil engineering, damage to buildings is mainly caused by movements that occur due to the settlement, swelling, and dynamic effects on clay soils. The type of soil movement depends on its physical and chemical properties, mineralogical structure, and textural properties. Clay soils are hydrophilic and their volume increases when in contact with water, and they shrink when water is lost. This volume change causes great damage to buildings built on clay soil if no precautions have been taken (Holtz & Kovacs, 1981). Chemical stabilization is one of the methods applied to improve clay soils. The aim of chemical stabilization is to change the properties of soil by adding various chemical additives, such as traditional ones like cement, lime, fly ash, and bitumen. In recent years, unconventional additives like enzymes, resins, and polymers have also been used as chemical stabilizers (Tingle et al., 2007; Anagnostopoulos, 2015; Mirzababaei et al., 2017; Kolay & Dhakal, 2020). Unconventional commercial stabilizers have been scientifically studied in recent years due to their different chemical structures and unknown interactions with the soil (Kolay et al., 2016; Kolay & Dhakal, 2020). Tingle et al. (2007) performed a study on clay soils improved with polyethylene vinyl acetate copolymer to better understand the physical and chemical bonding phenomena associated with unconventional stabilizers. Naeini et al. (2012) increased the strength of the clay soil by adding a cationic liquid polymer to soft clay. Anjaneyappa & Amarnath (2013) stated that liquid polymer added in different amounts to clayey–sandy soil increased its bearing capacity. Anagnostopoulos et al. (2014) investigated the mechanical properties of silty soil by using two types of water-soluble epoxy resins and stated that there was an improvement in strength parameters depending on the amount of resin. Hamidi & Marandi (2018) stated that it would be necessary to use new additives to increase the strength parameters of soft clay soils. They used cement and epoxy resin for the stabilization of soft clay soils and indicated that epoxy resin increased the strength parameters. Nowamooz (2016) examined the effect of polyurethane on soil stabilization by adding it to high-plasticity clay and revealed that the polyurethane additive significantly increased the soil resistance to load. Saleh et al. (2020) stated that they stabilized marine clay with polyurethane and polyurethane increased its shear strength. Similarly, Bak Sen et al. (2021) revealed that polyurethane was an

excellent stabilizer that increased the strength of soft marine clay. Acrylic polymers are used to improve the mechanical properties of soils (Ghasemzadeh et al., 2020). Additionally, Rezaeimalek et al. (2018) stated that the swelling pressure of clay, which was stabilized by using a styrene acrylic-based liquid polymer, decreased.

In this study, for preventing the negative effects of clay soils located in the road infrastructure, caused from freezing- thawing cycles, at first geotechnical properties of high-plasticity bentonite and low-plasticity kaolin clays were determined. After, liquid polymers (polyurethane, epoxy resin, and styrene acrylic) were added to bentonite and kaolin clays at 5 wt.%, 10 wt.%, 15 wt.%, and 20 wt.% of the optimum water contents. The effect of liquid polymers on the strength properties of bentonite and kaolin clays was then investigated. Unconfined compression tests and exposure to freezing-thawing cycles were performed on the clay samples with the liquid polymer additives, and the results were compared with the test results of additive-free clay samples.

1. Materials and methods

1.1. Clay minerals

In the experiments, the bentonite (B) used in the study was obtained from a commercial company and prepared as a dry, ground-up material by using bentonite clay deposits from Çankırı in Turkey. The natural calcium bentonite clay is high-plasticity (CH) clay. Similarly, the kaolin clay (K) used in the study was obtained from a commercial company, also prepared as a dry, ground-up material by using kaolin clay deposits from Balıkesir in Turkey. The kaolin clay is low-plasticity (CL) clay. The natural moisture contents of the clays could not be determined because of obtaining from a commercial company in dry state. According to the hydrometer analysis, the passing percentage of kaolin and bentonite clays from 0.075 mm sieve is 100%. The swelling pressure tests (ASTM D 4546) showed that the swelling pressure values of kaolin and bentonite clays were 9.3 kPa and 190 kPa, respectively. The chemical contents of bentonite and kaolin clays were determined by X-ray fluorescence (XRF) spectrometry (see Table 1). The geotechnical properties are provided in Table 2.

Table 1. Chemical contents of bentonite and kaolin clays

Constituents	Bentonite	Kaolin
SiO_2	61.28	69.10
Al_2O_3	17.79	15.20
Fe_2O_3	3.01	0.20
CaO	4.54	0.10
Na_2O	2.70	0.03
MgO	2.10	–
K_2O	1.24	11.07
SO_3	–	4.29
L.O.I.	7.34	–

Table 2. Geotechnical properties of bentonite and kaolin clays

Geotechnical properties	Bentonite	Kaolin
Grain size < 0.002 mm, %	30	28
Grain size < 0.075 mm, %	100	100
Specific gravity	2.73	2.63
Liquid limit, %	459	49
Plastic limit, %	87	26
Plasticity index, %	372	23
Soil classification*	CH	CL
Optimum moisture content, %	30	25
Maximum dry unit weight, kN/m^3	11.9	13.2
Unconfined compressive strength, kPa	283	307
Swelling pressure, kPa	190	9.3

Note: *Unified Soil Classification System (USCS)

1.2. Liquid polymers

Within the scope of this study, commercial polyurethane (P), epoxy resin (E), and styrene acrylic (S) liquid polymers were used. Polyurethane is a polymer made up of organic units connected to each other with urethane bonds. These macromolecules are formed by the reaction between a diisocyanate monomer or a polymeric isocyanate and a hydroxyl functional monomer in the presence of suitable catalysts and additives (Panda et al., 2018). Polyurethane properties include high strength, low viscosity, low density, and short gelling and curing times (Saleh et al., 2018). The epoxy resin used in this study was Bisphenol-A

type diluted with aliphatic glycidyl ether. Generally, epoxy resin shows good mechanical strength (Kimura et al., 1998). Additionally, toughness and rigidity of bisphenol A epoxies are high (Pham & Marks, 2005). With mixing the epoxy resin and hardener components, the product gains high strength and durability (Anagnostopoulos, 2015). Its viscosity is between 600 and 1200 MPa, while its specific gravity is 1.14. Styrene acrylic is acrylic resin cross-linked with polyisocyanates. Its specific gravity is 1.04.

1.3. Sample preparation

Liquid polymers were added to bentonite and kaolin clays at 5 wt.%, 10 wt.%, 15 wt.%, and 20 wt.% of their optimum water content, without changing the total water content of the samples (Saygılı, 2018). After mixing the clay and polymer, water was added and mixing was continued in a mechanical mixer (Gilazghi et al., 2016). After achieving a homogeneous mixture, the samples were cured for 1 and 7 days under laboratory conditions and then tested. Polyurethane was used with a hardener/polyurethane ratio of 0.132 suggested by the manufacturer, while epoxy resin was used with a hardener/epoxy resin ratio of 0.5, again specified by the manufacturer. The amounts of clays, polymers and hardeners used while obtaining the additive-free samples and the samples with additives are shown in Table 3.

1.4. Experiments

Unconfined compression tests (UCS) were carried out on both additive-free clays and the samples with additives in accordance with the ASTM D 2166 standard. Cylindrical specimens with dimensions of 35 × 70 mm were obtained from the samples compressed using a Harvard miniature compactor test instrument at standard Proctor energy. Three specimens were cured in laboratory conditions for 1 day and three specimens were cured for 7 days that were then subjected to unconfined compression tests. Specimens prepared in the same way and cured were wrapped in aluminium foil and subjected to 5 cycles of freezing-thawing. At the end of the cycles, unconfined compression tests were performed. In the freezing-thawing experiments, samples were frozen at –20 °C and thawed at 25 °C. One freezing-thawing cycle was completed in six hours (Ghazavi & Roustaie, 2010; Akbulut & Zaimoğlu, 2019). The unconfined compressive strengths and unconfined compressive strengths after freezing-thawing cycles of additive-free clays were determined without curing.

Table 3. Mixture compositions

Sample	Clay, g	Water, g	Hardener, g	Liquid polymer		
				Polyurethane, g	Epoxy resin, g	Styrene acrylic, g
B	120	36	–	–	–	–
K	120	30	–	–	–	–
BP5	120	34.2	0.21	1.59	–	–
BP10	120	32.4	0.42	3.18	–	–
BP15	120	30.6	0.63	4.77	–	–
BP20	120	28.8	0.84	6.36	–	–
KP5	120	28.5	0.17	1.33	–	–
KP10	120	27	0.35	2.65	–	–
KP15	120	25.5	0.53	3.98	–	–
KP20	120	24	0.7	5.3	–	–
BE5	120	34.2	0.6	–	1.2	–
BE10	120	32.4	1.2	–	2.4	–
BE15	120	30.6	1.8	–	3.6	–
BE20	120	28.8	2.4	–	4.8	–
KE5	120	28.5	0.5	–	1.0	–
KE10	120	27	1.0	–	2.0	–
KE15	120	25.5	1.5	–	3.0	–
KE20	120	24	2.0	–	4.0	–
BS5	120	34.2	–	–	–	1.8
BS10	120	32.4	–	–	–	3.6
BS15	120	30.6	–	–	–	5.4
BS20	120	28.8	–	–	–	7.2
KS5	120	28.5	–	–	–	1.5
KS10	120	27	–	–	–	3.0
KS15	120	25.5	–	–	–	4.5
KS20	120	24	–	–	–	6.0

Note: B: Bentonite; K: Kaolin clay; P: Polyurethane; E: Epoxy resin; S: Styrene acrylic; 5/10/15/20: Liquid polymer ratios

2. Results and discussions

2.1. Unconfined compressive strength test results

The unconfined compressive strength tests were conducted on the additive-free bentonite and kaolin clays and polyurethane, epoxy resin and stryene acrylic added bentonite and kaolin clays. Figure 1 shows the change in the unconfined compressive strengths of additive-free clays following the addition of polyurethane to bentonite and kaolin clays. When the samples obtained by adding polyurethane to bentonite and kaolin clays were cured for 1 day, the unconfined compressive strength increased between 23% and 59%, and between 17% and 61%, for bentonite and kaolin clays, respectively. When the curing time was 7 days, the increased rates were between 31% and 94%, and between 34% and 70%, for bentonite and kaolin clays, respectively.

Figure 2 shows the change in the unconfined compressive strengths of additive-free clays with the addition of epoxy resin. When 1 day of curing was applied to the bentonite and kaolin samples with epoxy resin, the unconfined compressive strengths increased between 5% and 31%, and between 8% and 25%, for bentonite and kaolin, respectively. With 7 days of curing, the increase rates were between 11% and 41% and between 18% and 32%. The unconfined compressive strengths of both bentonite and kaolin clays increased with an increase in the amount of polyurethane and epoxy resin additives.

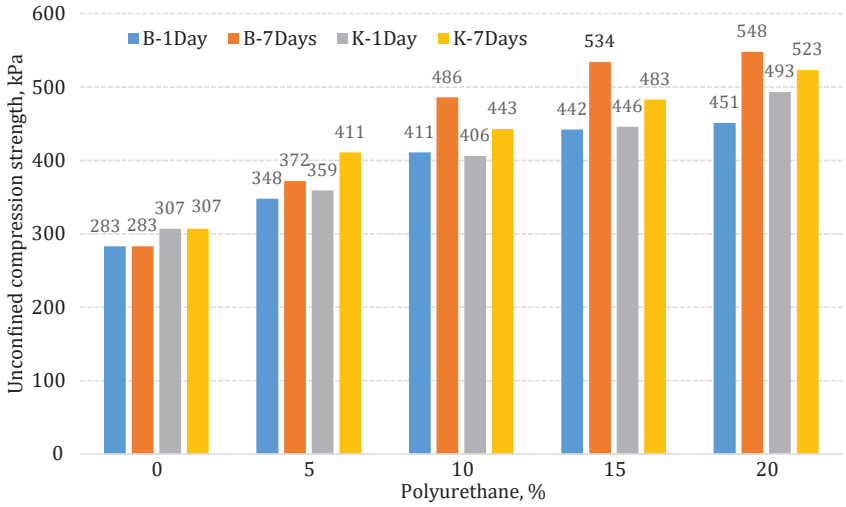


Figure 1. The change in unconfined compressive strengths of additive-free clays and clays with added polyurethane

Figure 3 shows the change in the unconfined compressive strengths of additive-free clays with the addition of styrene acrylic. It was observed that the styrene acrylic additive that was added to the bentonite clay at 5%, 10%, 15%, and 20% increased the unconfined compressive strength of the additive-free bentonite. The unconfined compressive strengths of bentonite samples with added styrene

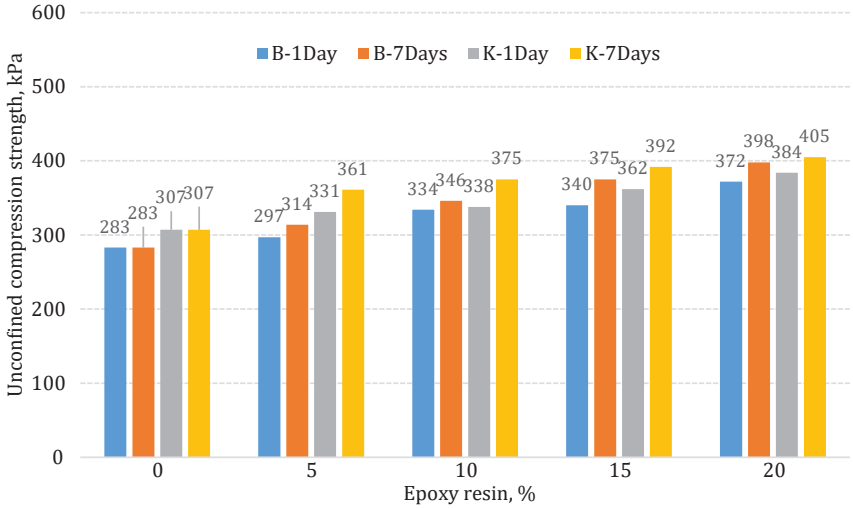


Figure 2. The change in unconfined compressive strengths of additive-free clays and clays with added epoxy resin

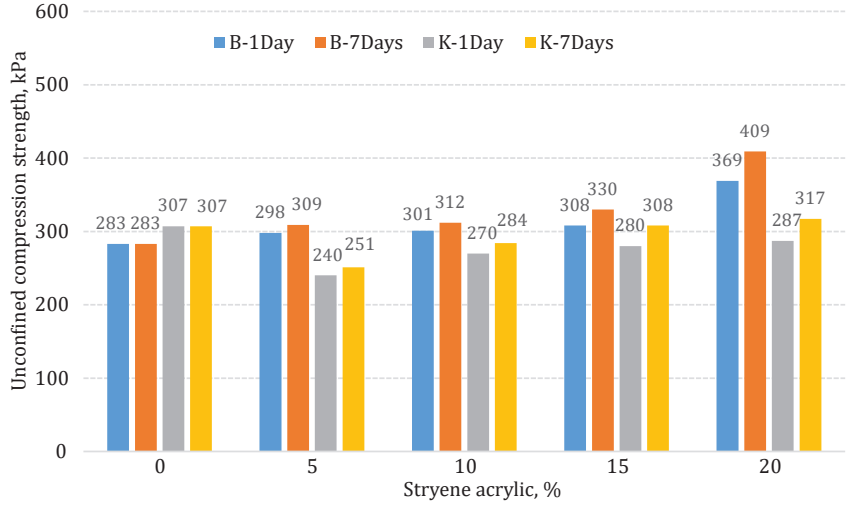


Figure 3. The change in unconfined compressive strengths of additive-free clays and clays with added styrene acrylic

acrylic and cured for 1 day increased between 5% and 30%, while the unconfined compressive strengths of bentonite clays cured for 7 days increased between 9% and 44%. For the kaolin clay, in contrast, the unconfined compressive strength decreased with the addition of styrene acrylic when compared with the additive-free kaolin clay. In the kaolin samples, the unconfined compressive strengths of samples cured for 1 day decreased by 7% to 22%, compared to additive-free clay. The unconfined compressive strengths of samples cured for 7 days with 5% and 10% additive decreased by 18% and 8%, while for the samples with 15% and 20% additive, there was a slight increase from 1% to 3%. It can be said that the styrene acrylic additive generally decreased the unconfined compressive strength of low-plasticity kaolin clay compared to the additive-free clay, and increased slightly it beyond a 15% additive level in 7-day cured samples. The disadvantage of kaolin when used as adsorbent is that it easily causes agglomeration (Şenol et al., 2022). For this reason, the cross linked polymer stryene acrylic may not have interacted with kaolin clay because of agglomeration and this may have reduced the strength of kaolin clay.

The unconfined compression tests showed that the unconfined compressive strength values obtained after curing samples for 1 day were lower than the unconfined compressive strengths obtained after curing for 7 days (Isik, 2019). According to the unconfined compressive strength test results, it is clear that polyurethane increased the strength of clays more than other liquid polymers. The high strength property of polyurethane could be the reason of high unconfined compression strength of clays (Saleh et al., 2018).

2.2. Freezing and thawing

The unconfined compressive strengths of samples after exposing them to 5 cycles of freezing-thawing are given in Table 4. Figure 4 and Figure 5 show the unconfined compressive strengths before and after the freezing-thawing cycles of bentonite and kaolin samples, respectively.

Table 4. Unconfined compressive strengths of samples after freezing-thawing cycles

Samples	Unconfined compressive strength after freezing-thawing cycles. Cured for 1 Day, kPa	Unconfined compressive strength after freezing-thawing cycles. Cured for 7 Days, kPa
B	239	–
K	286	–
BP5	337	345
BP10	365	374
BP15	375	383
BP20	397	486
KP5	311	342
KP10	342	369
KP15	407	432
KP20	453	488
BE5	270	287
BE10	288	296
BE15	291	306
BE20	304	322
KE5	297	307
KE10	308	315
KE15	314	327
KE20	343	359
BS5	283	302
BS10	277	299
BS15	279	306
BS20	320	376
KS5	225	228
KS10	240	248
KS15	251	269
KS20	269	303

Examining Table 4, when the bentonite and kaolin clay samples with added polyurethane, epoxy resin, and styrene acrylic were exposed to 5 cycles of freezing-thawing, it was observed that the unconfined compressive strengths of samples cured for 7 days were higher than those of samples cured for 1 day. When comparing the unconfined compressive strengths of samples before and after

freezing-thawing cycles, the samples subjected to freezing-thawing cycles had lower unconfined compressive strengths. Figures 6–11 show failure mechanisms of the bentonite and kaolin clay samples with additives after unconfined compression strength tests and unconfined compression strength tests after freezing-thawing cycles.

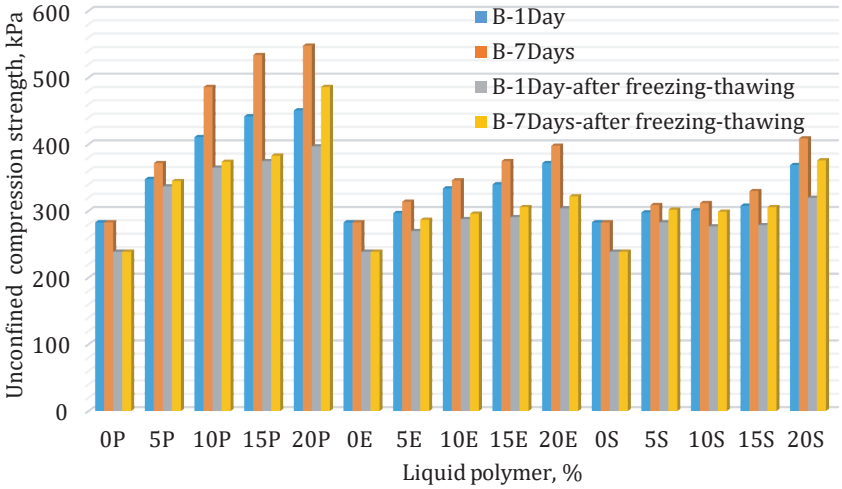


Figure 4. Unconfined compressive strengths of bentonite samples cured for 1 day and 7 days before and after cycles of freezing-thawing

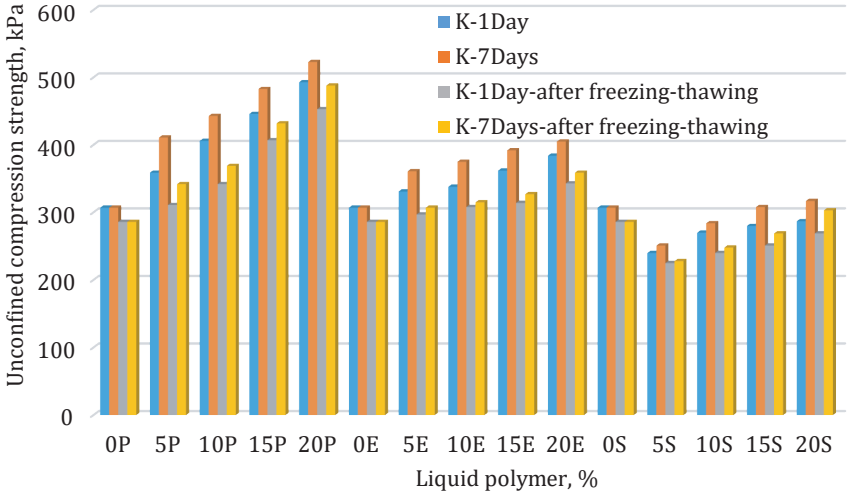
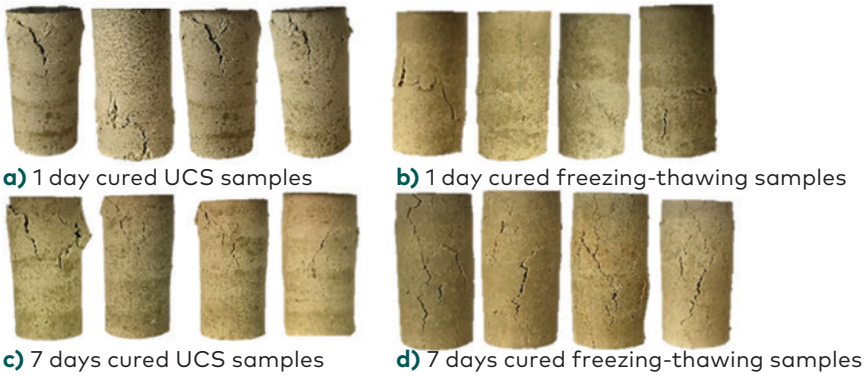


Figure 5. Unconfined compressive strengths of kaolin clay samples cured for 1 day and 7 days before and after cycles of freezing-thawing

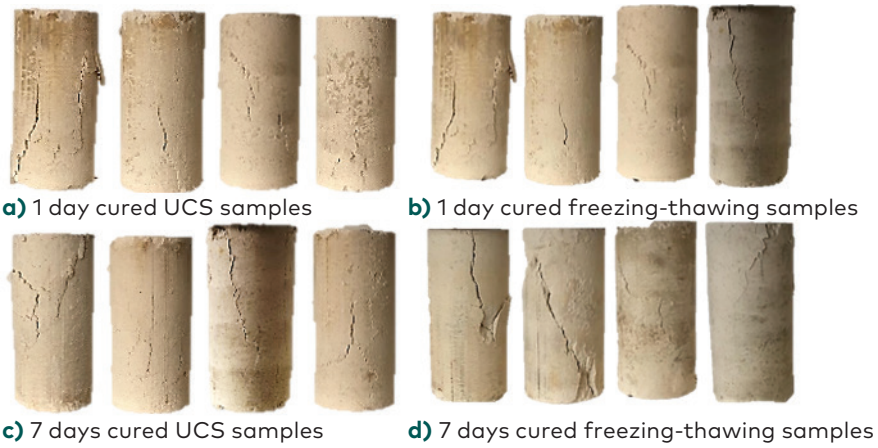
When Figures 4 and 5 are examined, it is seen that there was an improvement in the unconfined compressive strengths of bentonite and kaolin clay samples with polyurethane, epoxy resin, and styrene acrylic additives exposed to 5 cycles of freezing-thawing, when compared to additive-free clay. If the curing time was increased from 1 day to 7 days, polyurethane and epoxy resin increased the unconfined compressive strength of clays subjected to freezing-thawing cycles. It can be stated that polyurethane and epoxy resin additives improved the unconfined compressive strength of bentonite and kaolin clays subjected to



a) 1 day cured UCS samples **b)** 1 day cured freezing-thawing samples

c) 7 days cured UCS samples **d)** 7 days cured freezing-thawing samples

Figure 6. Failure mechanisms of bentonite samples with poylurethane (5%, 10%, 15%, 20%): a, c) after unconfined compression strength; b, d) unconfined compression strength after freezing-thawing cycles

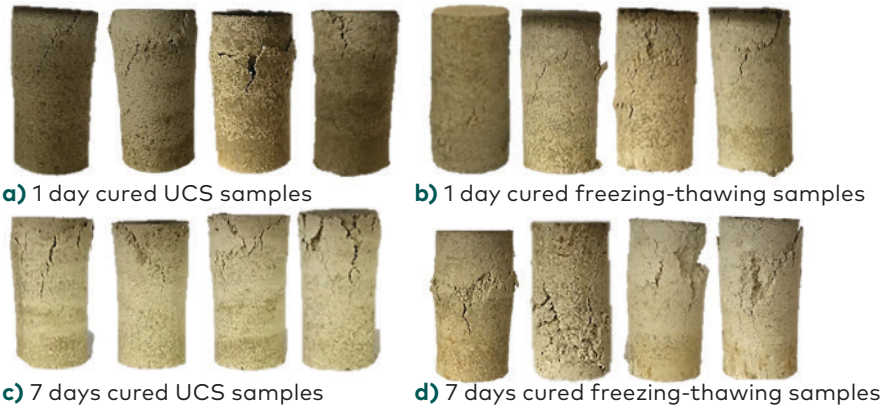


a) 1 day cured UCS samples **b)** 1 day cured freezing-thawing samples

c) 7 days cured UCS samples **d)** 7 days cured freezing-thawing samples

Figure 7. Failure mechanisms of kaolin clay samples with poylurethane (5%, 10%, 15%, 20%): a, c) after unconfined compression strength; b, d) unconfined compression strength after freezing-thawing cycles

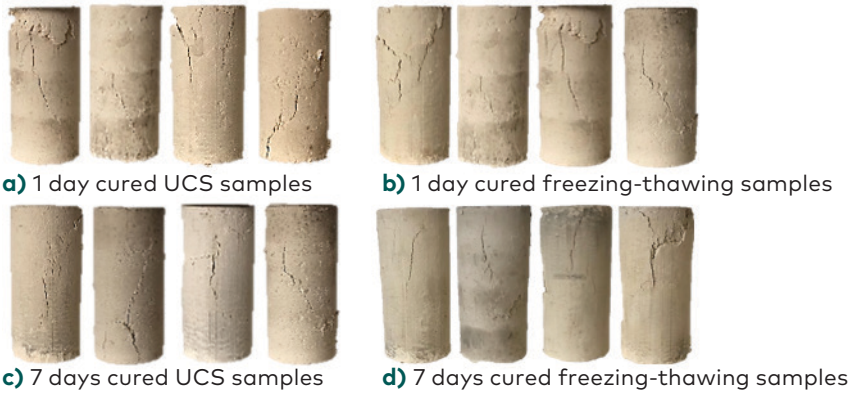
freezing-thawing, and a longer curing time had a positive effect on the unconfined compressive strength of those samples. Similarly, Güllü (2015) obtained that the strength of clay stabilized with lignosulphonate type superplasticiser of post-freeze-thaw strength showed positive results. It is thought that the freze-thaw cycles stabilized the strength of soil skeleton. This can be the result of the adhesion forces that occurred between clay particles because of the colloidal form of liquid polymers (Shi et al., 2021). The clay particles could gain a stable structure with polyurethane and epoxy resin additives.



a) 1 day cured UCS samples **b)** 1 day cured freezing-thawing samples

c) 7 days cured UCS samples **d)** 7 days cured freezing-thawing samples

Figure 8. Failure mechanisms of bentonite samples with epoxy resin (5%, 10%, 15%, 20%): a, c) after unconfined compression strength; b, d) unconfined compression strength after freezing-thawing cycles

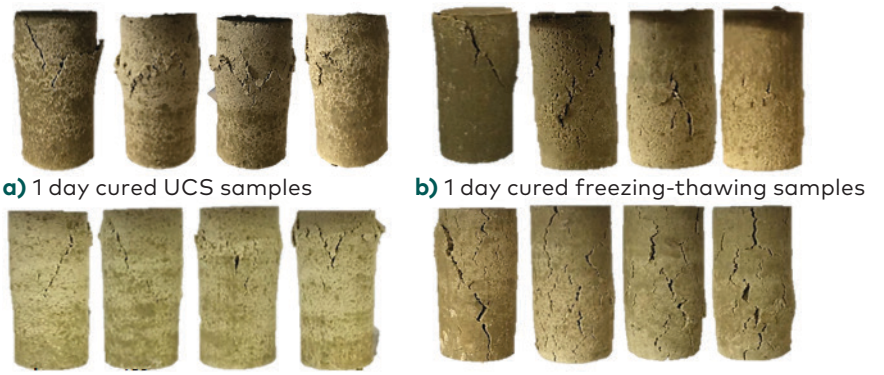


a) 1 day cured UCS samples **b)** 1 day cured freezing-thawing samples

c) 7 days cured UCS samples **d)** 7 days cured freezing-thawing samples

Figure 9. Failure mechanisms of kaolin clay samples with epoxy resin (5%, 10%, 15%, 20%): a, c) after unconfined compression strength; b, d) unconfined compression strength after freezing-thawing cycles

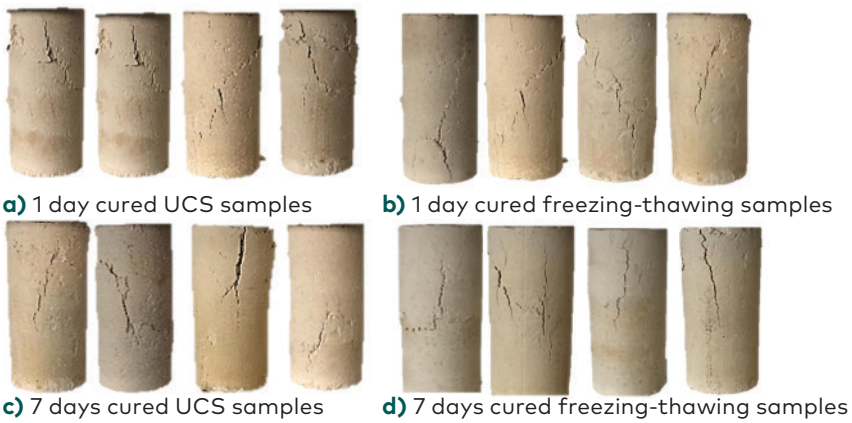
It was observed that the unconfined compressive strength of the bentonite samples with added styrene acrylic subjected to freezing-thawing increased in comparison with that of the additive-free bentonite subjected to freezing-thawing. Styrene acrylic added bentonite samples showed similar properties with polyurethane and epoxy resin added bentonite samples. However, when the results for kaolin clay were examined, it was determined that the styrene acrylic additive decreased the unconfined compressive strength of the sample subjected to freezing-thawing cycles in comparison with that of additive-free clay.



a) 1 day cured UCS samples **b)** 1 day cured freezing-thawing samples

c) 7 days cured UCS samples **d)** 7 days cured freezing-thawing samples

Figure 10. Failure mechanisms of bentonite samples with stryene acrylic (5%, 10%, 15%, 20%): a, c) after unconfined compression strength; b, d) unconfined compression strength after freezing-thawing cycles



a) 1 day cured UCS samples **b)** 1 day cured freezing-thawing samples

c) 7 days cured UCS samples **d)** 7 days cured freezing-thawing samples

Figure 11. Failure mechanisms of kaolin clay samples with stryene acrylic (5%, 10%, 15%, 20%): a, c) after unconfined compression strength; b, d) unconfined compression strength after freezing-thawing cycles

The decrease in the strength after freezing-thawing cycles of kaolin clay with styrene acrylic additive could be the reason of agglomeration because of the cross linked structure of styrene acrylic (Şenol et al., 2022). It is thought that because of the agglomeration, styrene acrylic could not diffuse in the kaolin clay and adhesion forces could not occur. In addition, the strength of kaolin clay subjected to freezing-thawing cycles increased when the amount of styrene acrylic additive was increased.

Conclusions

This study investigated the changes in the unconfined compressive strengths of bentonite and kaolin clays prepared with added polyurethane, epoxy resin, and styrene acrylic at 5%, 10%, 15% and 20%, based on curing samples for 1day and 7 days, as well as subjecting them to freezing-thawing cycles. The obtained results are given below:

- The unconfined compressive strength of bentonite and kaolin clay samples increased with increased amounts of polyurethane and epoxy resin.
- The addition of styrene acrylic to bentonite clay increased the unconfined compressive strength. In contrast, the unconfined compressive strength of kaolin clay decreased compared to additive-free kaolin clay with an amount of styrene acrylic up to a certain percentage, and beyond that, the unconfined compressive strength started to increase.
- It was determined that the unconfined compressive strengths of bentonite and kaolin clay samples with added polyurethane, epoxy resin, and styrene acrylic all cured for 7 days were higher than those of samples cured for 1 day.
- Polyurethane increased the unconfined compressive strength of clays more than epoxy resin or styrene acrylic.
- The unconfined compressive strengths of bentonite and kaolin clay samples with added polyurethane and epoxy resin exposed to 5 cycles of freezing-thawing were higher than those of additive-free bentonite and kaolin clay samples. Longer curing times had a positive effect on the unconfined compressive strength of samples after the freezing-thawing cycles.
- It was observed that the unconfined compressive strength of bentonite samples with added styrene acrylic that were subjected to freezing-thawing cycles increased compared to that of additive-free bentonite subjected to freezing-thawing cycles. Styrene acrylic added to the kaolin clay decreased the unconfined

compressive strength of the additive-free kaolin clay subjected to freezing-thawing; however, with an increasing percentage of styrene acrylic, the post freezing-thawing unconfined compressive strengths increased.

As a result of the study, it was seen that liquid polymers increased the unconfined compressive strength of bentonite and kaolin clays. Moreover, liquid polymers generally had a positive effect on the unconfined compressive strengths of bentonite and kaolin clays subjected to freezing-thawing cycles. Based on these results, it can be stated that liquid polymers can be used to improving the strength behaviour of clay soils and can be used as stabilizers especially for soil improvement in cold climate zones exposed to freezing and thawing.

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