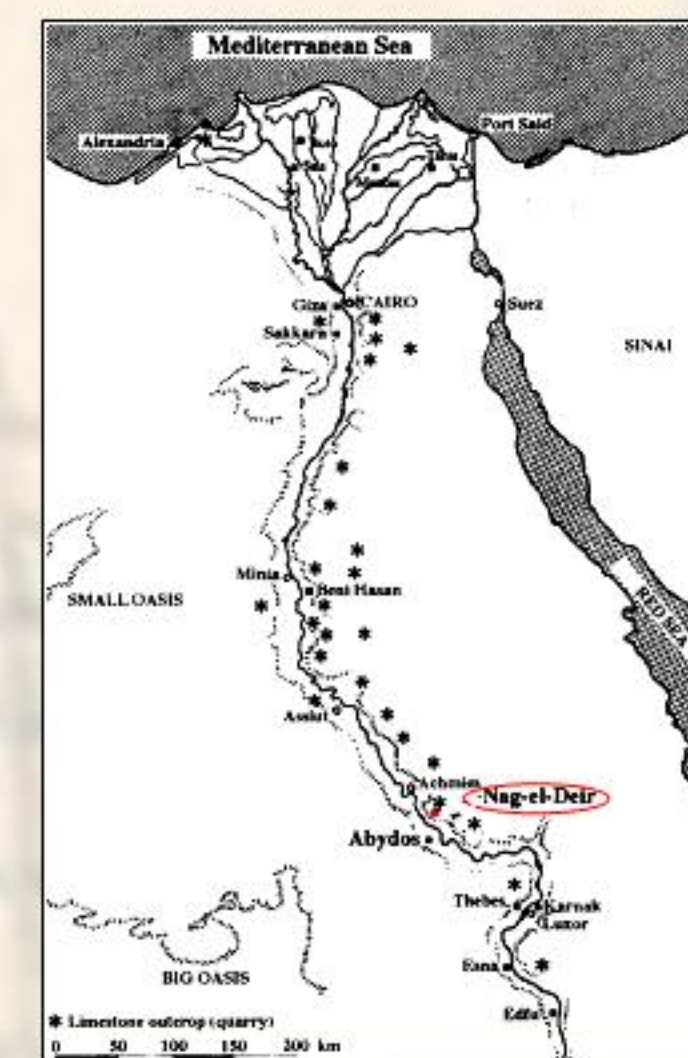


# Diaminoalkanes as Swelling Inhibitors in Ancient Egyptian Limestone Conservation

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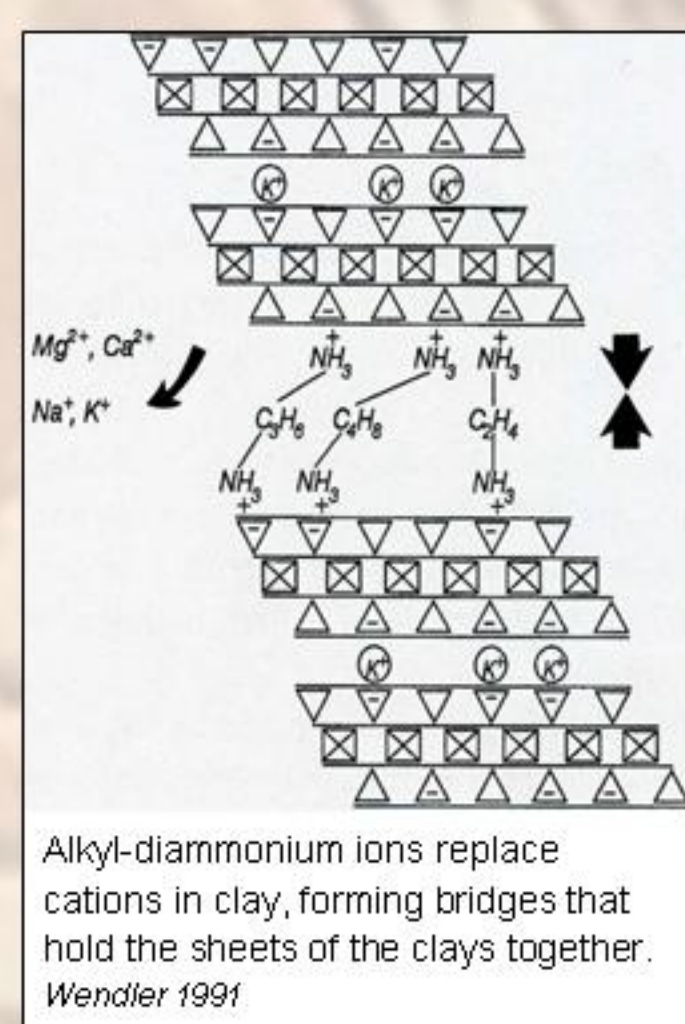
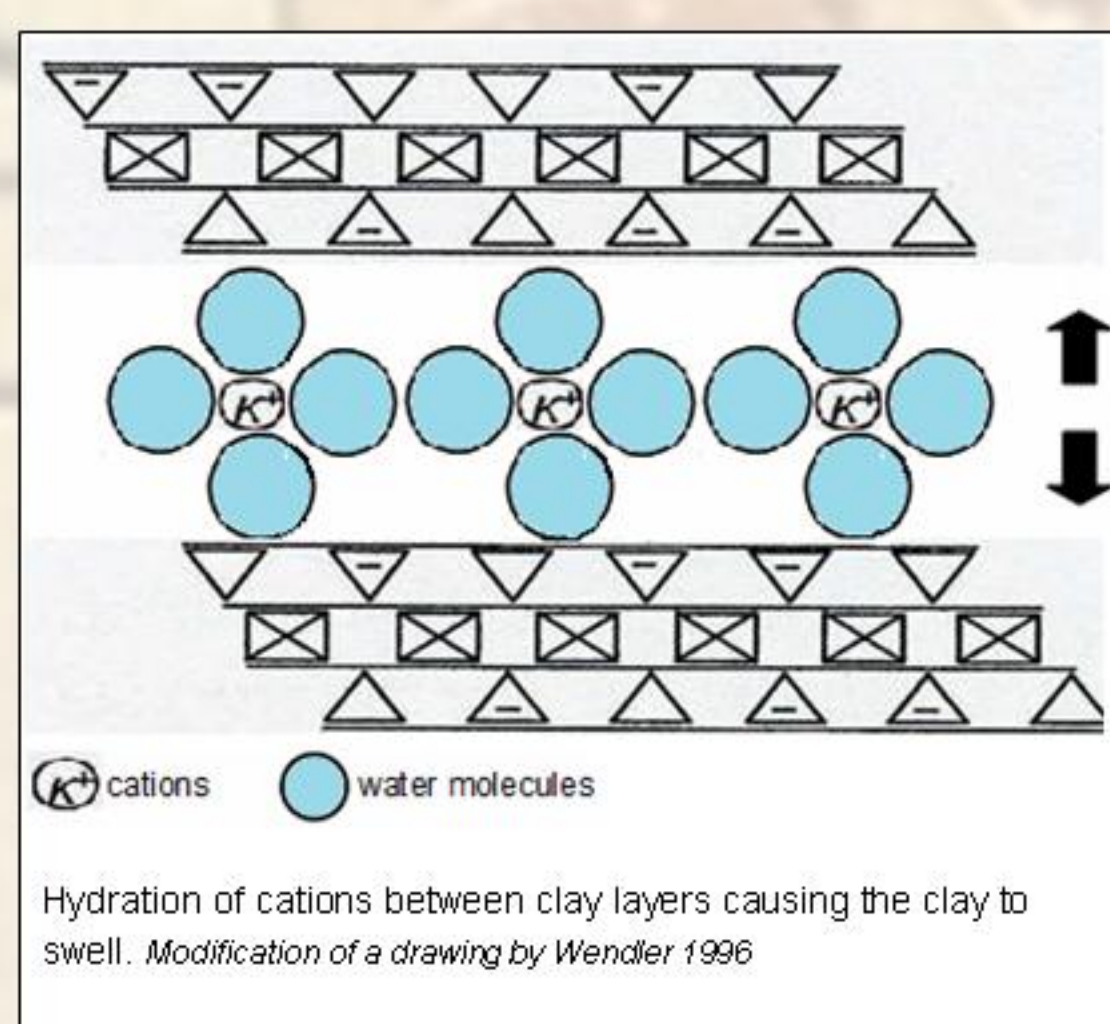


## Abstract

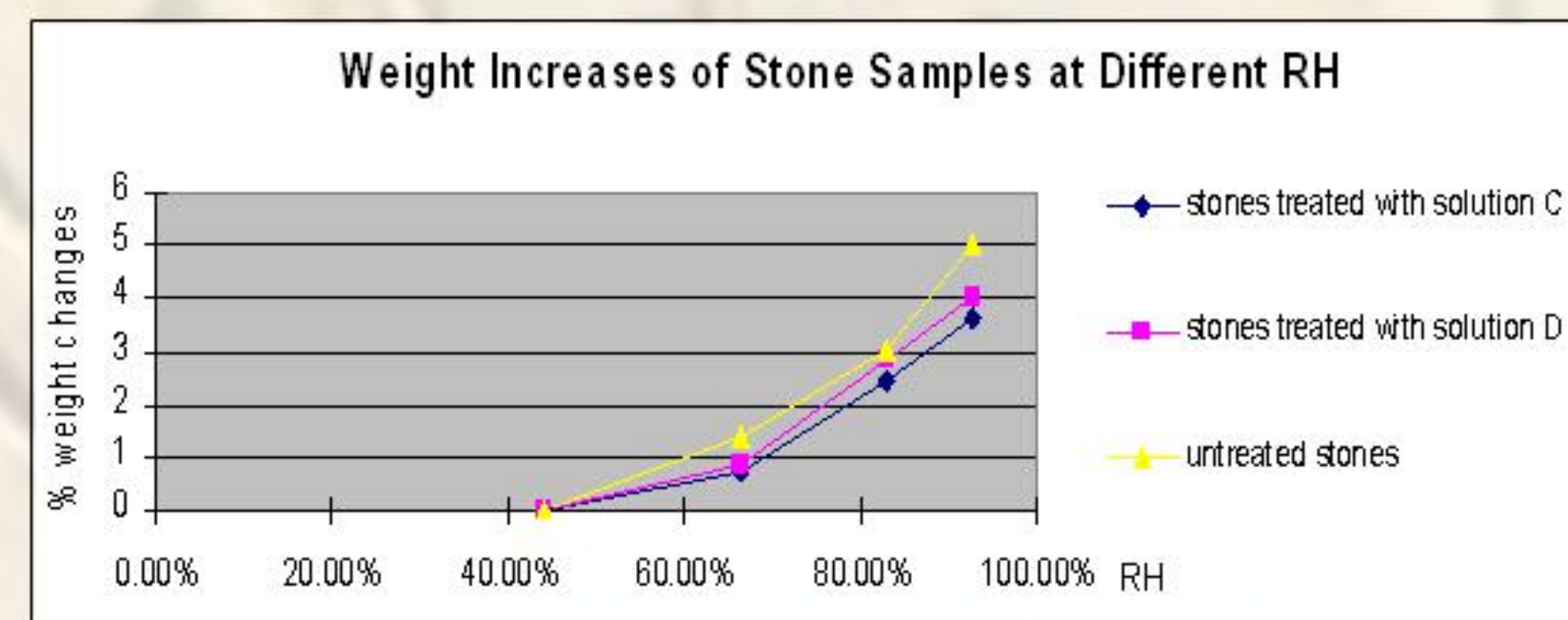
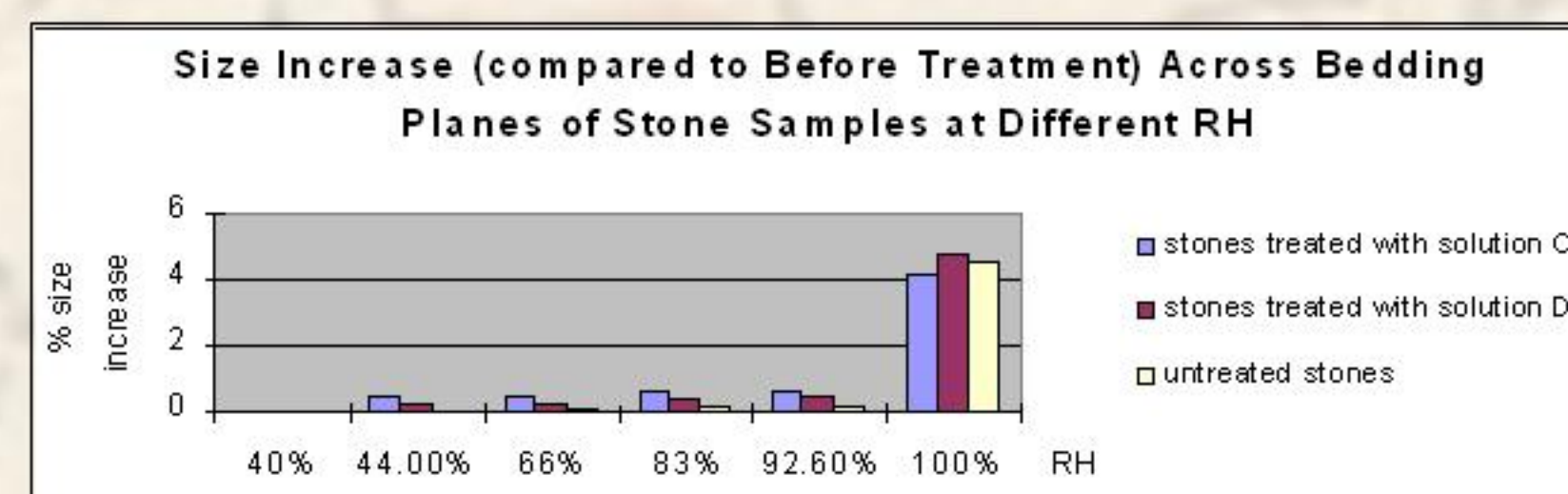
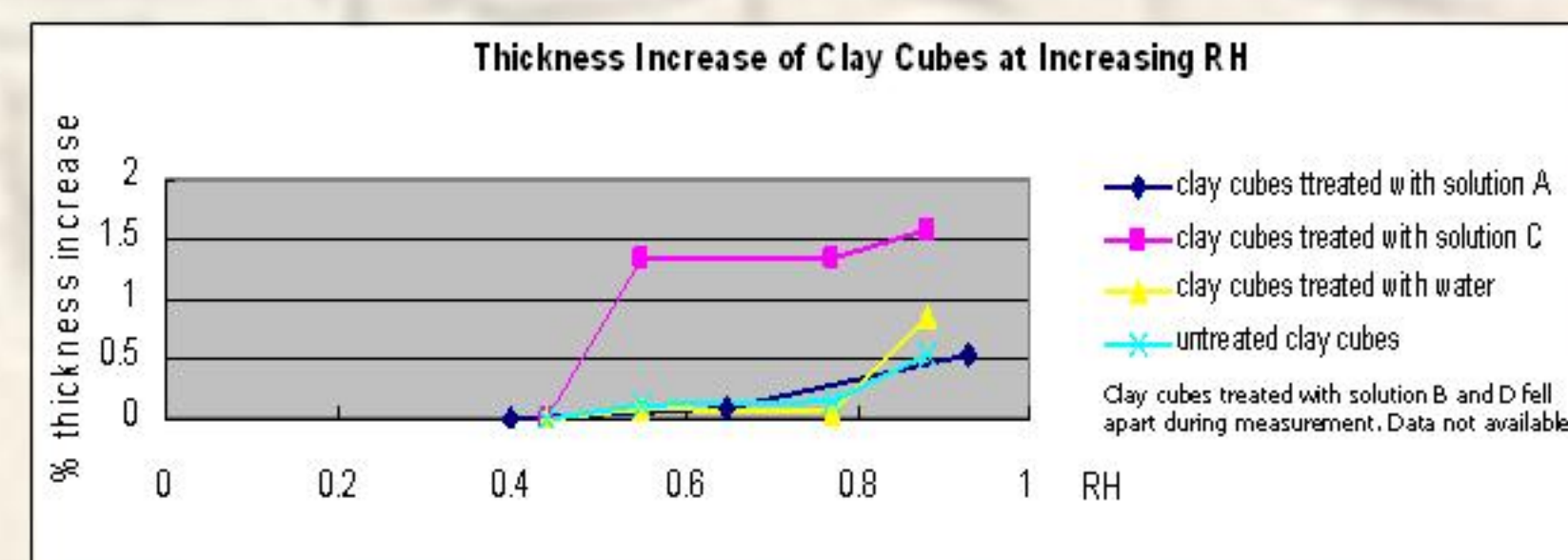
Limestone sculptures from Nag-el-Deir found in different museums exhibit ongoing deterioration during their storage in museum environments due to the cyclic expansion and contraction of the clay content. In lab situations, diaminoalkane surfactants show up to 50% swelling reduction of sandstone and tuff. The objective of this research was to examine the swelling reduction effect of diaminoalkanes on clay-bearing Egyptian limestone and to assess this treatment. Solutions of 1,4-diaminobutane (DAB) and 1,4-diaminobutane dihydrochloride (DABHCl) were tested. The treated and untreated samples were evaluated for: their expansion and water absorption under different relative humidities; damage resistance to RH fluctuations; as well as swelling, colour change, and salt formation due to treatment.

## Introduction

- The clays sepiolite and palygorskite concentrating along bedding planes of limestone swell when in contact with water. This causes the stone to expand in the direction perpendicular to the bedding planes, resulting in delamination, fracture, spalling and flaking of stone sculptures
- Swelling of the clays is caused by hydration of exchangeable cations between layers of negatively charged clay surface
- Diaminoalkanes  $[H_2N-(C-H_2)_n-NH_2]$  reduce swelling of clay minerals by replacing the hygroscopic cations between clay layers with its protonated amine ends  $[^+H_3N-(C-H_2)_n-NH_3^+]$
- Amines are protonated when diaminoalkanes are neutralized with an acid



## Results



## Experimental

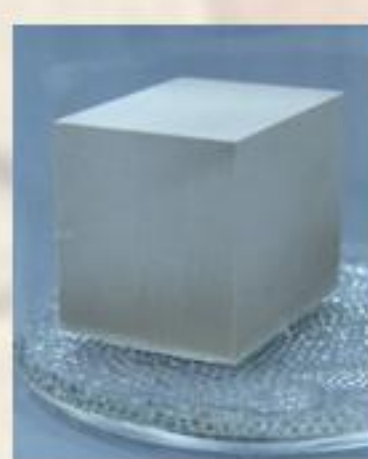
### Materials

- Clay cubes of sepiolite and palygorskite mixture (9:1 w/w): slightly moistened clay mixture pressed into cubes using hydraulic press under 2-ton pressure
- Nag-el-Deir limestone: from a Second- to Fourth-Dynasty cemetery, provided by Phoebe Hearst Museum of Anthropology, Berkeley, CA
- 0.25M Surfactants:

Solute	Solvent
A DAB neutralized with glacial acetic acid	Deionized water
B DAB neutralized with glacial acetic acid	50% ethanol in deionized water
C DABHCl	Deionized water
D DABHCl	50% ethanol in deionized water + 1 drop of HCl

### Methods of application

- Each sample placed on a layer of fine glass beads in a beaker with their bedding planes perpendicular to the bottom of the beaker, which was sealed with Parafilm.
- Surfactant was added slowly with a pipette until it just touched the bottom of the sample, which took up solution by capillary action until saturation.
- Samples left to dry in the fume hood for two days
- Due to toxicity of solution A and B, they were only used to test on clay cubes and eliminated later on from the experiment.



### Methods of Evaluation

- Swelling behaviour of treated and untreated samples evaluated by percentage of their size increase in the direction perpendicular to bedding planes at increasing RH followed by soaking
- Resistance to RH fluctuations evaluated by subjecting the stone samples in 12-hour cycles between 20% and 65% RH. The weight loss of each sample due to spalling and flaking was recorded after 50 cycles
- Colour changes of the stone samples due to the treatment were measured using Minolta hand-held colourimeter
- Soluble salts content of treated and untreated stone samples analyzed with ion chromatography (IC) by Queen's University Analytical Services Unit

## Conclusion

### Effectiveness of DAB and DABHCl in swelling reduction

- DAB neutralized with acetic acid rendered greater reduction in swelling of clay cubes than DABHCl
- For the surfactants, water was a better solvent than 50% ethanol in water in terms of their effectiveness in swelling reduction
- After two hours of soaking, swelling of the stone samples treated with DABHCl in water was 10.8% less than the untreated stone samples

### Drawbacks of treating the limestone with diaminoalkanes

- Treatment caused 2-3% expansion in the direction perpendicular to bedding planes. Fissures along bedding planes during treatment were observed on some stone samples
- Expansion due to treatment was lessened when using 50% ethanol in water as a solvent for DABHCl; however, DABHCl in 50% ethanol was proven to be less effective than DABHCl in water
- The water uptake abilities of the samples were slightly reduced after the treatment
- The treatment caused slight discoloration to the treated stone
- Amines are hazardous and can cause bad reactions to some people

To sum up, diaminobutane treatment is not a suitable approach for the preservation of Nag-el-Deir limestone sculptures. Expansion of the stone samples during the treatment is greater than expansion due to increasing RH. Reduction in swelling of the treated stones, by 10.8%, was observed only when the samples were soaked in water.

## Acknowledgments

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