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Submitted as a partial fulfillment of the requirements for degree of Bachelor of Science in Ceramic Engineering at Missouri School of Mines and Metallurgy.

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Approved
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During the procedure of testing Ozark Plastic Fire clay in 1928-29 a few of the test bars were unintentionally left in a laboratory drier and after some months it was found that these bars were decidedly disintegrated into a powder without any dnown special treatment. This phenomena was then duplicated by Mr. Paul who describes the experiment as follows:

Raw Ozark Clay (of the same lot as used in the weathered and unweathered clay experiment in 1927-28) was used for this work. The clay was already ground to pass a 20 mesh sieve. The clay was tempered, and four 6x1x1" bars were moulded. The bars were properly dried at 225°F.

After two days at 225°F., the bars showed no cracking and they were placed in the small drier operating at 175-185°F. After two months, three bars showed very small hairline cracks on the surfaces. After three months at the above temperature, all the bars showed the cracking to a greater extent than on previous esamination.

After 3-1/2 months no perceptible surface change is noticeable from previous examination. Probably the cracking is developing into the bar itself.

After 6 months the cracks became enlarged and longer.

After 7 months (at the same temperature in the drier) the bars were so disintegrated that theyfall to pieces upon the slightest touch.

In order to properly investigate the cause of this phenomena it was decided to repeat the experiment under similar conditions so that no error could enter the investigation which would cause any deviation from the results previously obtained from storage in the department's warehouse, and this particular clay is thought to have been in storage for about a year which in most probability is from the same shipment as the clay which previously produced the unusual phenomenom. The clay was charged through a Blake jaw-crusher and then ground by means of a Stutevesant double roll crusher. The clay was then screened through a 20 mesh screen and the residue on the screen was passed through a Braun pulverizer with the blades set at maximum separation to prevent excessive pulverization and only moderate grinding. In this way all the material selected from quartering was used in the bodies later made from this clay. A screen analysis made from this clay. A screen analysis made on this sample was as follows: 35%, 20-40 mesh; 17% 40-60 mesh; 14% 60-80 mesh; 20% 80-200 mesh; 13% through 200 mesh.

From comparative data on screen tests made on materials similar to Ozark clay which was ground under the same conditions in this laboratory the above results indicated a normal grinding treatment and it was therefore supposed that the clay sample used possessed normal grain size and was similar to the sample which showed the phenomenom already mentioned. The clay was then dampened with water to the extent of about 15%, andthen allowed to stand over night. The clay was tempered and worked to the best working consistency and it was then molded in brass molds in the form of bars 6x1x1".

Forty bars were made this way and then marked for shrinkage with 5" marks, and also for identification the plastic weights were obtained and recorded. The bars were placed upon wooden pallets and allowed to stand in the room atmosphere which was normal for the fall of the year. The bars were turned twice a day to prevent warping,and after two days a few of the bars were weighed and then reweighed every day to determine their constant weight and thorough air drying. After five days the bars showed constant weight (weighing to .05 gram), and they were then placed in the laboratory drier which is subject to themostatic control and the temperature was set at 225°F. After two days the bars were removed and their dry length and dry weight was determined and recorded. The bars were then placed in the smaller drier and the temperature was adjusted at about 80-90°C, and the air was allowed to circulate in the drierby opening the draft vents specially provided for that purpose. Two bars were drawn as trials every week and placed in the dessicator to cool. They were then weighed with an allowable error of + or - .02 grams. Also the length was measuredand the specimens carefully inspected for any signs of cracking or other weaknesses. After this the bars were broken across a 5 inch span by means of adding lead shot to a bucket suspended from the center of the test bar. The modulus of rupture was calculated and recorded for all specimens.

This procedure was strictly followed throughout theexperiment in order to prevent error in the results inherent in other methods. After the bars were broken the edges of the bars were crushed with the hand because it was thought that ay slight weakening due to the drying phenomenon only, would be most apparent on the edges because there is more surface area exposed per unit depth and breadth than in any other part of the bar. The bars so tested were brushed to remove any loose adhering particles, relabeled, and weighed to plus or minus .02 garms and then placed back in the small drier. Thred bars were taken from the drier each week for the tests just described. After a month or two since no changewas apparent in any of the physical properties investigated only two bars were drawn as trials per week with with the hope that enough bars could be kept in the drier to last about eight months. The drier used could hold only about 75 bars.

In the beginning about twenty-five bars were made from the same sample of clay only after agine the clay for a week. These bars were conviently marked and placed in the drier with the bars made from unaged clay. The purpose of this was to gain comparative data of the aged and raw clay , and to determine the effect, if any, upon the anticipated disintegration of the clay in question. It was thought that the aging would develope any colloidal content of the clay we should expect the results to vary in the aged and raw clays. Thes samples were likewise drawn as trials along with the standard raw clay bars to afford immediate detection of variation in any of the properties investigated.

In order to further investigate the effect of the colloidal material in the clay upon its physical properties it was attempted to destroy a part of the colloidal material by the addition of lime to the clay and then molded in standard bars and then dried. The distributionof the lime w.s poor and in making tests upon the bars it was found that in general the properties of the body was not greatly changed and in breaking the bodies fracture occured in segregated lime lumps and the modulus of rupture was very low and had no value. This experiment was further abandoned. If the lime could be uniformly mixed so that it would have maximum effect on the colloidal material, results of a different nature would be expected, although there is no reason to believe that it wouls have any effect upon self disintegration in the drier.

After six months of observation and testing with no indication of weakening orother phenomenon, it was decided to vary the conditions in drying of the clay to possibly effect some of the phenomena looked for. It was supposed that a severe heat treatment might cause some changes in the clay body which might partially account for any disruption of the clay was supposedly subject to only the drier heat and air circulation. If by any chance the original clay had been subjected to severe drying temperatures by the readjustment of the drier temperature by some person other than the observer, who did not reveal the change, the same disintegrating effect should be produced under severe treatment under control. About thirty bars were made of the original Ozark clay by the same method in making the first bars used in this investigation. They were marked for identification and shrinkage and their plastic weights taken for calculation of the water of plasticity.

The bars were then air dried to constantweight for about 3-4 days and were ready for the test. In order to gain a comparison of thebehavior underthe conditions of the test to be run, another clay body was selected andmade to bars just as with the Ozark clay. Empire clay from A. P. Greene F. B. Co., was selectedas it was the only fire clay with any degree of plasticity available. While this clay was of the semi-flint variety and lacked the plasticity a theOzark clay it might be expec, a to show similar changes under severe heat treatment. About fifteen each of the clay bars were placed upon perforated pallet plates in such a way as to give free circulation offair, These pallets were then placed in the large laboratory drier with thermostatic control and with the temperature at about 210°F. The following heating schedule was followed curing the 12 hour test.

1:20 - 210°F	8:00 - 350°F
2:40 - 250°F	11:00 - 375°F
3000 - 300°F	2:00 - 400°F

Four barswere taken from the drier at the following temperatures; 200°, 290°, 305°, 400°, and the remainger of the bars were allowed to remain during the cooling. The bars when taken from the drier were placed in a dessicator and allowed to cool. Their dry length, weight and modulus of rupture were then found and also a close inspection was made of thesamples for any apparent changes.

Discussion of Results: This investigationwas begunby me without having previously observed the phenomena.Before I could attempt to account for the causes or reasons, I deemed it necessary to account or reproduce this phenomena.

Since this experiment was carried over a period of approximately eight months, and under the conditions stated in the first part of this paper, there has been no appreciable change in the physical properties of the clay to even approximate the results as stated by the first observer of this phenomena. As this experiment did not reproduce the disintegration of this particular clay body over this period of time much room is left for conjecture as there were no clear cut facts brought out in this investigation. Since all the data accumulated through this investigation have nearly constant values I think it will be necessary only to include the results over periods of a month for comparison.

Month	1	2	3	4	5	6	8
M. R.	354	374	346	358	348	350	354
% Shrink							
W. P.	22.1						
Loss in Max. weight	<.5 gr.	<.04 gr.					

From this data it is clearly shown that there was no apparent disintegration or weakening in the clay body for there is a maximum deviation of only 8% in the average monthly modulus of rapture values. The fact that the greatest deviation results during the first month of drying may have some significance in that, it is supposed that nearly all the hygroscopic water is removed during that period and a consequent setting of the grains with relation to each other may be of such a nature as to effect greatest attraction and therefore greater body strength. The decrease on strength after this peak may be attributed to shrinkageof the grains themselves which would necessarily decrease the cohesive force of the body. However, after the first month the M. R. values are practically constant.

It was attempted in this experiment to weig. the bars used, with an accuracy of $\pm$ or - .04 grams, but it was found that this small permissible error was too small to give dependable results without accounting for factors as, humidity, loss, of weight, loss of weight of material from handling, etc. However, there were no noteceable weight losses throughout the test except within the first month and then only to the extent of slightly less than 0.5 grams. This loss was undoubtedly due to the removal of hygroscopic water which remained after air drying and was occluded in the very fine pores of the clay. The reason for attempting this method of detecting loss of weights during drying was to obtain any indication dehydration of minerals during this test which might cause disintegration of the body due to the consequent reduction in volume of materials when dehydrated.

In the test using lime as a destroying agent of the colloidal material in the clay, the method was abandoned because the lime was not uniformly distributed and the results obtained were indefinite. I do not believe that this method of investigation is of value because it is very improbable that small amounts of organic colloids would cause a break down of the body in which it is included.

The test which was run with the object of varying the temperature in the drying of this clay probably the only part of this experiment which furnished the observer with any definite data. It was previously intended to run this test according to a prearranged heating schedule, but it was difficult to obtain the desired temperature with the dial control. Bars were placed at various parts of the dryer in order to obtain all possible conditions which might exist in this dryer. The trials drawn at the temperatures mentioned in the procedure of this experiment were treated much in the same way as the bars from the small drier. There was practically no loss of weight in any of the bars drawn throughout the test and it is supposed that no dehydration of the minerals was effected by the temperatures reached in this test. The linear shrinkage apparently did not change from the air dried values.

The modulus of rupture values obtained on the trial bars is given in the table below. It is interesting to note the increase in strength with the early period of effective drying. The strength increases with the time and temperature of drying up to 320°F. in the case of the Ozark clay and the Empire semi-flint follows the same trend. This phenomenon is similar to that observed in the original bars in the small dryer. It may be said that the strength of these types of clays increases above that of air dried clay by virtue of further removal of hygroscopic water by artificial means. The same reason as suggested heretofore may be likewise applied in this case.

	200°F	250°F	290°F	320°F	400°F
Ozark clay (K series)	201	300	369	384	354
Empire semi-flint clay	118	---	178	156	182

Two trials of Ozark clay bars that had been in the small drier for nearly 7 months were also used in this test and allowed to remain in the drier through the maximum temperature and cooled slowly in the drier. The average modulus of rupture for these bars was 320 pounds which is easily within 15% deviation from the maximum M. R. values found in this experiment. This fact clearly proves that the eight months treatment and this special treatment has not affected the stength of the clay in question. However, all the bars whose values have been given were either placed in a dessicator or allowed to cool slowly in the drier. Three bars of the Ozark clay were removed from the drier at about 410^{o}F and then placed in a natural draft of air through a window, where they were allowed to cool. The next day upon inspection it was found that several small cracks had developed on the edges and ends of the bars which had been most efficiently exposed to the cooling air. One of t e bars was immediately used for the modulus of rupture test but the calculated value of this test was 322 pounds. This showed that no general weakness had developed and the cracks were probably only on the surface of the trial. However, within two weeks the cracks in these air cooled bars had developed greatly and parts of the surface were loose and could easily be removed by slight pressure with the fingers. The cracks have no general direction, but they are most abundant along the edges and ends of the bars. These bars will be kept for future observation to determine whether or not disintegration will continue along these cracks. The first observer of this phenomenon declares that these cracks were similar to thebody originally observed and if this is the case it might be expected that the bars will follow the same process of disintegration. If this is true it might be assumed that the thermal shick of this hightemperature produced the necessary first weakening of the body when later develops throughout the body. This is only a conjecture which does not actually exist as yet, and can only be verified on the future by observation after this paper has been written.

All important question of why this type of clay has at one time exhibited self-disintegration under the stated conditions, and has failed to reproduce the phenomenon under the same comditions during this investigation still remains unanswered and numerous unaccounted-for conditions could be supposed, but all with no reason of belief. For instance, it is possible that the Ozark clay which first showed this phenomenon may have been selected from a different part of the clay pit than the sample of clay tested in this investigation. Or, the bars in the original experiment may have been subjected to excessive heat treatment or other effect which would cause disintregation, and these facts were unknown to the observer who first reported this phenomenon. Also, it the bars were at any time wetted with excessive moisture this would cause slaking of the clay which in its effect in very similar to the effect of eidintregation originally described.

Since the development which was wxpected, has failed to show itself in this period of eight months I believe that further experimenting under the same donditions is of no value and should be dropped.

BIBLIOGRAPHY

The Effect of Lime on Certain Cracking Clays

N. B. Davis -- Trans. Am. Cer. Sac. XVII Page 497

In Orton's work on seeking a remedy for the cracking of clays, he used various chemical coagutants as HCl, NaCl, Etc., but in each case these were reported ineffective and impractical. Preheating the clay to a temperature at which part of the structure was destroyed made such clays workable and satisfactory.

Reasoning from the effect of lime on plastic loils in effecting an open soil, these experiments were based upon the addition of varying amounts of lime to the clays in question.

It was found that the gelatinous matter causing excessive plasticity and shrinkage is of an inorganic nature in one case and of organic nature in another. With different clays it was found that three percent lime, in one case, corrected the drying difficulty while as much as twelve percent lime was required for another clay. It requires at least ten percent of lime to appreciably attack silicic acid. Magnesia is more effective than lime.

In the cracking clays, the substance causing the trouble is the very finely divided matter which has a great affinity for water. Lime also has a great affinity for water. By getting an intimate mixture of clay and lime water and "clay" Matter and conditions are favorable to the immediate formation of lime salts with the acid silicates and silicis acids.

In practice it would be necessary to add the lime in a dry state.

Strength of Clay Bars as Influenced by the Temperature of Dying

C. H. Kerr and R. J. Montgomery -- Journal. Am. Cer. Soc. XV Pg.352

Over 100 percent variation in the modulus of rupture of raw clay bars was caused by variations in the drying treatment after the bars have reached the air-dried condition.

Test bars dried at temperatures below approximately 100°C do not attain full strength.

If air-dried bars are placed directly in an oven at temperatures above 100°C., thay are disrupted by the rapid escape.of water vapor. For a reliable and satisfactory test, bars after being air-dried must be dried to constant weight at a temperature safely below 100°C and then dried to constant weight at a temperature somewhat above 100°C.