

## Clay as Thermoluminescence Dosimeter in diagnostic Radiology applications

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### Abstract

**Background:** As part of efforts to isolate and utilize local and naturally occurring materials for development of thermoluminescence dosimeters and other technologies, an earlier report had shown that Nigerian clay showed prospects of utility as a thermoluminescence dosimeter (TLD). This paper reports the investigation of the basic thermoluminescence properties of clay at x-rays in the diagnostic radiology range, including dose monitoring in abdominal radiography.

**Methodology:** Clay sourced from Calabar, Nigeria, was tested for thermoluminescence response after irradiation at diagnostic radiology doses, including application in abdominal radiography dose monitoring in a clinical setting.

**Results:** Results show that thermoluminescence (TL) output in natural clay is very low, but demonstrates enhanced performance with the addition of common salt. Specific TL characteristics of good repeatability for individual and batched pellets (variability index of 3.08%) and a high degree of trap emptying were observed. It had a glow curve peak at 275°C; with traces of spurious thermoluminescence emission at the reader anneal temperature. There was evidence of good batch homogeneity (< 30%) and a similar pattern of dose absorption in abdominal radiography with commercial Lithium Fluoride (LiF TLD-100). A high fading rate (over 30% in twelve hours) and low sensitivity (12 times less than LiF TLD-100) however, signal the unacceptability of clay as a TLD in diagnostic radiology in the forms studied.

**Conclusion:** Clay demonstrates poor TL response at diagnostic radiology doses. However, its water absorbing property offers a means of overcoming the hygroscopic nature of common salt. This could be explored to improve the use of sodium chloride as a radiation detector.

**Keywords:** Clay, Thermoluminescence, Dosimeter, Detector, Radiology, x-rays.

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### Introduction

Thermoluminescence (TL) has been known for a number of centuries during which period it has found wide application in many fields involving radiation detection and measurement. TL materials emit photons of light when heated after previous exposure to ionizing radiation. The number of photons emitted during the heating process is proportional to the radiation absorbed within the TL material. The intensity of this light at any temperature is a function of electron hole recombination occurring in the material during readout<sup>1, 2</sup>. The characterization of materials as dosimeters for different types of radiations and their optimization with different doping materials have been investigated<sup>3-6</sup>. The response of the different TL detectors to different radiation types under different irradiation conditions of energy, annealing and temperature have also been studied<sup>7-13</sup>. Characterisation of TLD materials is usually against standard criteria and recommendations by international and national regulatory bodies<sup>14-15</sup>.

In developing countries, thermoluminescence dosimetry is still very expensive and access to commercially available TL phosphors is limited. This, in addition to the high cost of TLD readers, has limited the radiation monitoring efforts in many areas. The need for a reversal of this position, coupled with the increasing demand for exploitation and harnessing of local and available raw materials for national development are the basis for the current study. Savings from local production of the TL material could be channelled into obtaining the TL readers.

Chemical analysis of clay from the eastern shores of Nigeria, show that it consists of 39.5% alumina, 46.54% silica, 13.6% water, in addition to traces of Fe<sub>2</sub>O<sub>3</sub>, MnO, Na<sub>2</sub>O, MgO, and K<sub>2</sub>O<sup>16</sup>. Samples from adjacent localities show a similar lower content of the alumina<sup>17</sup>. There are also varying concentrations of other mineral impurities like quartz, anastase, pyrite, feldspar, mica and montmorillonite. Individual clay deposits can be unique due to their mode of formation and physical properties of a given blend<sup>18</sup>.

A pilot study has shown that clay ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ), has a TL property. However, the investigation was limited to only the TL readout for technical reasons<sup>19</sup>. Besides, it was not known whether the samples used in the study were natural or not as they were obtained from the open market. Clay is sold sometimes as salted samples in the locality where the samples were sourced. Clay has been reported as a radiation shielding material<sup>20-21</sup>. However, there is no direct report of utility of clay in TL measurement.

This study examines some thermoluminescence characteristics of clay based detector. It seeks to establish the suitability of clay as a TL dosimeter for application in low dose measurements commonly encountered in diagnostic radiology. The success of the study would lead to the development of a new TL detector from the material. The prospects of economic gains that could arise from this in terms of employment opportunities and ultimately, overhead cost reduction in running radiation dose measurements in a developing economy are incentives for this study.

## Materials and Method

Commercial samples of clay obtained from the Watt market in Calabar, Nigeria were made into powder by crushing and grinding the blocks. 60 gram of the clay powder was measured into 21 beakers, one for natural clay and ten each for the two test solutions of salt. Salt solutions were made by dissolving Tidmann's<sup>®</sup> rock (RS) and table salt (TS) obtained from a superstore in water to make salt solutions of concentrations 0.86 mol/l, 1.71 mol/l, 2.57 mol/l, 3.42 mol/l, 4.28 mol/l, 5.13 mol/l, 5.99 mol/l, 6.85 mol/l, 7.70 mol/l and 8.56 mol/l of water. The clay powder was added to these solutions, while water was added to the beaker labelled natural clay. This arrangement allowed for the study of the TL performance of natural clay, and the variation due to the presence of salt. Excess solution was drained off and the mixture left to set.

The clay paste was layered onto the 50-hole aluminium mould to make pellets. Weights of the mould were taken before and after introduction of the clay, and thereafter, hourly while being oven dried at 60 °C, until there was no further change in weight. Pellets were made in a cylindrical shape with thicknesses of 1mm and a diameter of 3 mm. Pellet weights averaged 0.02 mg and only pellets not exceeding  $\pm 5\%$  of the mean weight in each group were selected for the study. This was done to reduce the effect of weight variation on the results.

## Initialisation, Exposure and TL analysis

A total of 200 pellets of clay were initialised as required for studying a dosimetric system<sup>22</sup>. Initial tests were carried out to ensure no loss of sensitivity by the initialisation process. They pellets were subjected to thermal anneal in a Pickford 879/A/1 model oven at the optimum temperature of 400°C for 1 hour. The temperature was chosen for giving the best results in pellets annealed in the 200–400°C range. Following this, the pellets were allowed to cool to room temperature over 24 hours. Initialised pellets were stored at room temperature and pressure.

Lithium Fluoride LiF TLD-100 (3.2 mm x 3.2 mm x 0.9 mm) chips from Harshaw Chemical Co, calibrated with known doses of x-rays monitored with a type 511 UNFORS<sup>®</sup> ionisation chamber with calibration traceable to national standards in the UK, were used with the clay pellets. This provided a means of comparison of clay TL performance.

A portable x-ray generator with a total inherent tube filtration equivalent to 2.5mm Al and calibrated for exposure from 0.03 to 5 s, with preset kVp of 60–100 and tube current of 10–30 mA, was the x-ray source for initialisation and TL tests. The reproducibility of the unit was accurate to  $\pm 3\%$  at 80 kVp, 60 mAs and 90 cm focus to detector distance (FFD). Exposure of samples was done with an incident x-ray dose of 13.4 mGy monitored with a type 511 UNFORS<sup>®</sup> ionisation chamber. TL response and transparency of natural clay was assessed. Salted samples were exposed and for TL response and to determine optimum salt concentrations. Following this, TL characteristics of dose response, repeatability, residual dose, batch homogeneity and fading. Laboratory and field comparison of test material performance with commercial TLD (LiF TLD 100) was made. Field comparison involved the use of clay pellets with LiF chips in plastic sachets with negligible x-ray absorption to monitor entrance surface doses (ESD) for 16 patients undergoing abdominal radiography. The exposures were made with a Siemens Multix Pro P unit (Siemens UK) with total filtration of 3.5mm Al. Reproducibility for the unit was better than 4% for all parameters. Mean abdominal exposure factors were 75kVp and 40 mAs (range 70–80 kVp and 38–56 mAs, respectively).

TL analysis was done with a model 654D Vinten<sup>®</sup> TOLEDO reader incorporating a pure liquid nitrogen flush/cooling system, and connected to a computer to obtain the TL intensity and temperature data, with which a glow curve was plotted. The timing and temperatures

for the readout cycle, based on trials to obtain maximum signal output was set at 16 seconds and 140 °C for pre-heat, 20 seconds at 280 °C for read and 12 seconds at 410 °C for anneal, respectively, at the heating rate of 25 °C/s.

### Results

TL response of clay obtained from the readout sessions are presented in the following section under the different variables studied.

### TL intensity

The plot of TL output of a material against temperature or time is the glow curve. The TL intensity is proportional to the concentration of either trapped charge or recombination centres<sup>2</sup>. Glow curves plotted in this study for both natural and (table) salted clay are shown in Figure 1. A major peak at about 265 °C (for the natural clay sample) at the heating rate of 25 °C per second is observed. The salted pellets displayed a curve (broken line) with higher TL intensity giving its main peak at between 230 °C and 260 °C. Two other peaks are observed around the pre-heat (at 160 °C) and anneal temperatures (at 410 °C) respectively. The pre-heat temperature peak occurs only with irradiation of the samples, and may be due to emptying of very shallow traps, while 410°C peak was consistent even in background readings, and could be due to spurious TL in the material, probably due to chemiluminescence<sup>22</sup>. The effects of pre-irradiation anneal conditions are a subject for further investigation.

Table I: TL results for repeatability in 10 pellets

Pellet No. 1	Exposure number/TL output per exposure							Pellet mean	COV
	2	3	4	5	6	7			
1	506	630	544	553	542	508	512	542.14	0.079
2	539	642	521	528	534	512	513	541.29	0.084
3	521	528	523	526	552	521	511	526.00	0.024
4	526	524	562	534	516	523	512	528.14	0.031
5	563	540	519	524	505	511	513	527.57	0.034
6	545	552	524	505	510	512	509	522.43	0.036
7	523	526	548	861	561	514	509	577.43	0.218
8	525	526	524	542	518	520	519	524.86	0.015
9	528	524	526	530	523	518	508	522.43	0.014
10	521	538	522	625	518	512	514	535.71	0.075
Batch mean	529.7	553	531.3	572.8	529.7	515.1	512	534.8	
Batch COV	0.029	0.081	0.028	0.186	0.032	0.009	0.006		

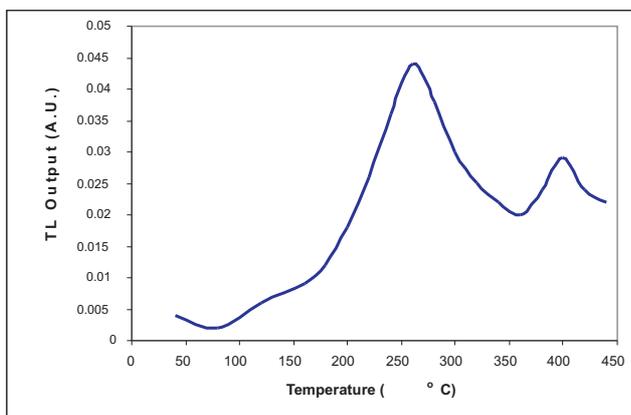


Figure1: Typical TL curve of natural clay

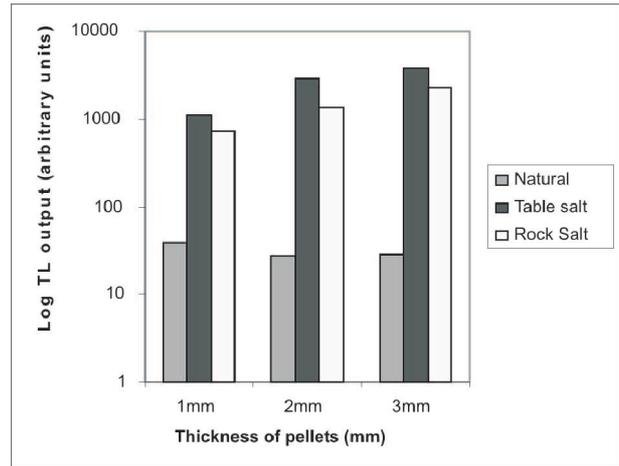


Figure 2: TL response by different thicknesses of the natural and salted clay samples

Samples of 3mm thickness produced the highest TL response but were dropped in the study because pellets were breaking in the TLD reader.

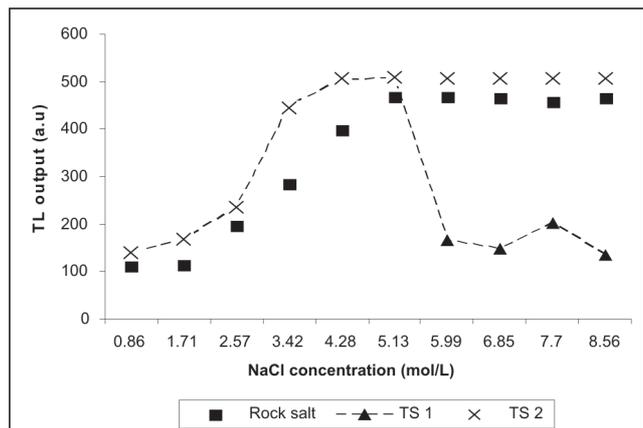


Figure 3: Variation of TL output with NaCl concentrations

TS1 are Table salt readings taken progressively one after the other. The drop is the result of time lapse between analyses of the pellets. This effect (fading) is prevented by taking all readings directly after irradiation (TS2 and RS).

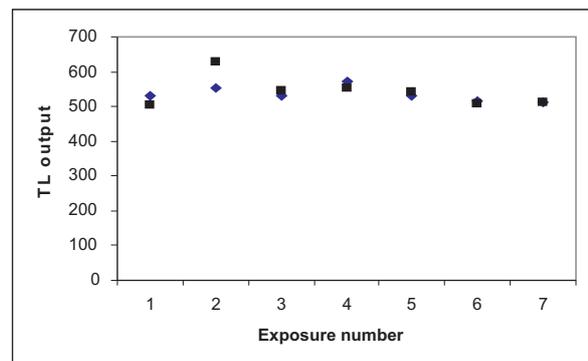


Figure 4: Plot of TL response for one pellet (●) and a batch of 10 pellets (○) over seven exposures.

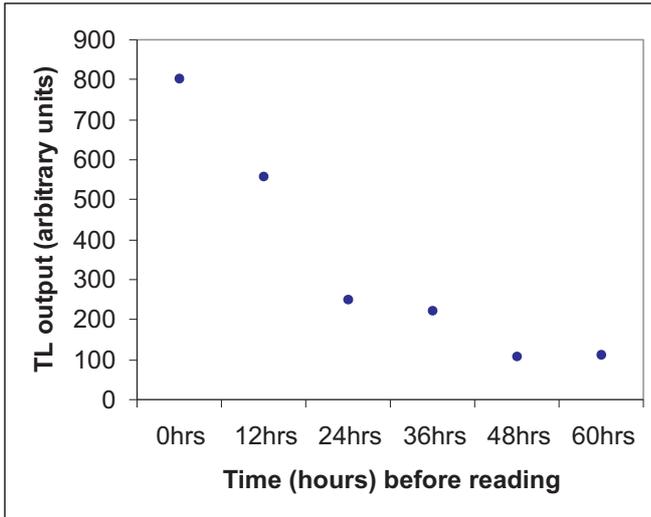


Figure 5: Fading of Pellet sample with highest TL output (TS), over time

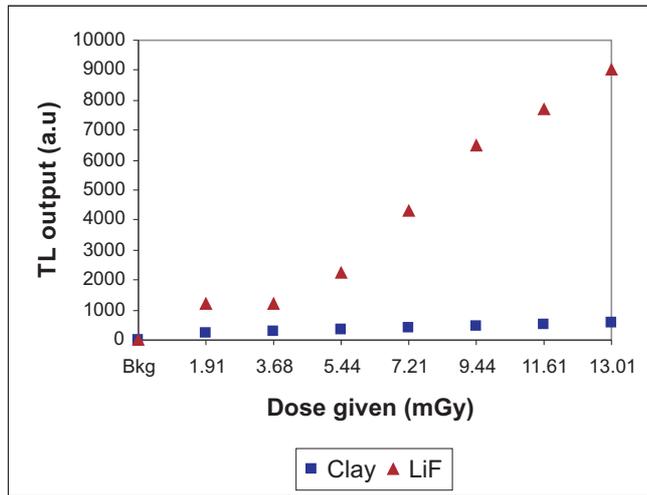


Figure 6: TL output of clay (■) with LiF (▲) using the same set of detectors and exposure conditions.

### Material transparency

To obtain the material's transparency, 10 pellets each were exposed to x-rays at energies between 60 and 95 kVp. For each energy, five pellets were analysed from the exposed side and the other five from the side distal to the x-ray source. The ratio of TL intensities from the two sets of pellets is plotted in Figure 2.

A mean transparency of about 95% was observed over the range of energies used. However, transparency increased with increasing tube potential up to about 80 kVp, where the ratio of TL intensity from the exposed and unexposed sides of the pellets appeared to be constant. The reduced transparency at low energies may be why clay has been recommended for shielding against secondary radiation.

### Variation of response with NaCl concentration

Ten concentrations in mol/Litre of rock (RS) and table (TS) salt were used in production of the pellets. Figure 3 shows the result of TL analysis for different salt concentrations and salt types. There was a rise in TL response with salt concentration up to a maximum at the concentration of 5.13 mol/l, for both table and rock salts. Beyond this point, the response plateaus despite further increase in concentration. There is evidence of table salt pellets showing a greater TL output than rock salt. This may possibly be due the effects of iodide present in table salt. The difference in TL output for the salt types is however not statistically significant ( $P < 0.05$ ).

### Dose response

A requirement of a TL detector is the ability to show a linear variation with dose. TS-C test samples were exposed to radiation doses ranging from 0.5 13.4 mGy. Doses were varied with a change in both kVp and mA since the x-ray source used had both parameters ganged together. The results were averaged over three pellets per dose. The standard deviation ranged from 0.6 to 4.4, being higher with dose.

The clay samples show a linear relationship with dose at doses below 1 mGy. Beyond this point the response becomes sublinear.

### Repeatability

This is a measure of the reproducibility of the TL output of each detector and of the batch. 10 pellets were subjected to seven rounds of anneal, irradiation, read and anneal. The TL output is given in Table I.

The coefficient of variability, defined as the quotient of the standard deviation and mean percent ( $SD/mean$ ) % of each pellet (intra-pellet variability) is less than 1%. For the batch of ten (ten) pellets tested over seven exposures, mean COV per pellet was 0.053 (SEM 0.02). This implies a high degree of consistency in the TL output when the same pellets are used for radiation detection over seven exposures (Figure 4). An index of variability for the detectors [DVI] given by

$$DVI = \frac{[(SVI)^2 - (RVI)^2]^{1/2}}{\text{mean TL}} \quad (1),$$

was defined, where SVI is the mean value of the % SDs of each pellet and RVI is the mean of the % SDs of each cycle of exposures<sup>22-23</sup>. From the TL values for 7 repeated exposures for 50 pellets (Table 1), DVI comes to 3.1%. The variation of the TL output with successive exposures for a batch of 50 (Fig. 5) was also better than 5%.

### The Residual Dose

The residual dose depends on the TL material, dose exposure and irradiation history of the individual detector<sup>13</sup>. To study the residual dose, 5 new detectors were chosen at random from the unused group within the batch being studied. These were annealed, irradiated at 13.4 mGy and read to obtain first time readings. They were then re-read immediately after to obtain the second readings. This procedure was repeated over twenty cycles. The ratio of the second and first readings for the five readings determined from their means was 0.0084 (range 0.0082 - 0.0089). This implies very good trap emptying by the samples.

### Batch Homogeneity

The IEC<sup>15</sup> recommends that the evaluated TL value for any one dosimeter in a batch should not differ from the value of any other in the same batch by more than 30. To study batch homogeneity, 48 detectors were annealed and irradiated at 13.4 mGy. TL read out values were studied and the mean of the maximum and minimum values determined for two exposures. Using the procedure described by Furetta and Weng<sup>22</sup> and Azorin et al<sup>24</sup> and, and equation 1:

$$\Delta_{\max} = \frac{(M - Mo)_{\max} - (M - Mo)_{\min}}{(M - Mo)_{\min}} \times 100 \leq 30\% \quad (1)$$

Where,  $\Delta_{\max}$  is the uniformity index of the batch, and M and  $M_o$  are the mean values for the maximum and minimum readings for the TL response of the batch.  $\Delta_{\max}$  was 9%. This meets the requirement as described in equation 2.

### Stability of stored signal

To study the fading property of the clay pellets, 50 pellets were exposed to the same radiation dose (13.4 mGy) and processed for TL output over a period of 60 hours. The results of this test actually determined the keeping time of the detectors. Exposed pellets were stored within the enclosed anneal tray immediately after irradiation, at room temperature and read at 12 hourly intervals. Stored TL signal decreased rapidly with storage time, with over 80% of the signal being lost within 2 days of sample exposure. This is not acceptable in practice. Figure 6 shows the rate of signal loss with time.

### Comparative response with Lithium Fluoride (LiF)

Laboratory comparison of LiF TL absorption with the clay samples was carried out using their respective response to dose. Results show wide differences in dose response (Figure 7a). A similar large difference was obtained in field measurements of abdominal radiography doses, as shown in Figure 7b. Abdominal exposure factors ranged

from 5.8 - 180 mAs and 70 - 81 kVp, for a focus to image receptor distance of 100 cm. All examinations were in the supine AP position.

The relative sensitivity (equation 3) of clay samples to LiF was calculated from exposure of equal weights of the two detectors to two doses (1.12 and 9.44 mGy).

$$R(D) = \frac{S(D)_{\text{testmaterial}}}{S(D)_{\text{referencematerial}}} \quad (3)$$

R(D) values of 0.17 and 0.07 were obtained at the doses used. This shows that the relative sensitivity of clay is dose dependent. Patient doses were varied largely by mAs (tube current and time) changes, although the kVp was a factor in a couple of patients. It follows from the above that LiF TLD-100 is on the average > 12 times more sensitive than the clay pellets.

### Discussion

The performance of the clay salt-pellets has shown dependence on salt concentration (Figure 3), energy dependent transparency (Figure 2) and linearity at low doses (Figure 4). A coefficient of variability with repeated exposures of 8% for a batch of 50 pellets, and 6% (range 1.4 - 21%) per pellet over seven exposures was also obtained. The calculated value of DVI which relates pellet performance to the number of exposure cycles shows a similar result. A DVI of 3.1% agrees with the individual pellet and batch repeatability values. All the values in this category are below the recommended limit of 10%. These results suggest the possibility of clay as a radiology dose detector.

The TL properties of clay could be understood and explained by the many defects in the material due to the presence of many impurities in clay. The major constituents of clay,  $Al_2O_3$  and  $SiO_2$ , have been reported to have good TL properties<sup>7,13</sup>. These form the bulk (over 85% by weight) of the compound with numerous impurities making up the remainder<sup>16</sup>. The sensitivity of a dosimeter is dependent on the mass of active phosphor in it<sup>25</sup>. The poor TL response of natural clay as seen from the outlined results suggests that the large presence of  $Al_2O_3$  and  $SiO_2$  does not add up to good TL performance. Usually, a small quantity of TL phosphor is required in a material to form the defect and region of high sensitivity which absorbs incident radiation and produces TL signal when heated.

To improve performance, doping is the typical option and has been used to enhance the TL properties of many detectors<sup>26</sup>. This option could not be pursued due

to unavailability of doping facilities. This is however a subject for further study. In this work, the addition of common salt to clay samples became the means of TL enhancement suggesting that the results obtained in Egbe et al.<sup>19</sup> were most probably due to use of salted clay samples.

Sodium Chloride, an alkali halide, is from the family of materials which has produced excellent TL detectors and has itself been mentioned as a TL detector<sup>27</sup>. Used both as single crystals and as a powder, sodium chloride shows a linear (up to 0.05 Gy) and supralinear TL response and has two glow peaks at 80°C and 220°C, with others at 500°C<sup>28</sup>. It also has great signal retention capacity of up to seven months, but is limited in use by its hygroscopicity and wavelength dependence sensitivity.

The TL output of clay samples in this study (Figure 1), is therefore attributed to the salt. The benefit of this clay-salt combination becomes the overcoming the hygroscopicity of NaCl by the water sorbing property of clay. This was evident in the high physical stability of the pellets despite exposure to air at room temperature. The pellets showed high resistance to handling and environmental factors. Physically, they required some substantial pressure to crush, which was not the case in the natural form. What is worth questioning in all these is why the combination could not produce enhanced signal retention capacity despite NaCl's high retention ability as reported by Kaibao et al.<sup>28</sup>. The fact that clay contains numerous substances and impurities suggests the presence of many competing shallow traps which may account for this. The method of production of the pellets

and the reader heating rate may also have an effect on the performance. It may be useful to explore some other production methods (for instance sintering) and both annealing and reader heating cycles and temperatures in order to draw more extensive conclusions. Studies of the materials self-absorption may also be useful.

## Conclusion

Nigerian clay has been tested for TL property for possible application in dose measurements in diagnostic radiology. The results show poor TL response for natural clay but enhanced output with salted clay. Good repeatability for individual and batched pellets (variability index of 3.1%) with high degree of trap emptying, good batch homogeneity, high fading rate and low sensitivity, characterize the samples. By these results, natural and salted clay cannot be used in monitoring radiation doses in diagnostic Radiology. However, the combination offers prospects for overcoming the hygroscopicity of NaCl, which had been a set back in its use as a radiation detector.

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