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Proton induced X-ray emission analysis of soils with various length of fallow: The effect on soil total elemental composition

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ABSTRACT

Fallowing of whatever kind is beneficial to elemental (nutrients) recovery. To what length (years) should a land be left fallowed for optimum elemental recovery is of serious concern to stakeholders owing to the limited availability of land. This work aimed at accessing the effect of fallow duration on soil total elemental composition. Proton induced X-ray emission (PIXE) technique has been used to assess the effect of relative longer length of fallowed land on soil total elemental composition using a 5SDH Tandem Pelletron accelerator available at Centre for Energy Research and Development, Obafemi Awolowo University Ile-Ife. The results at a 2.5 MeV energy revealed the presence of elements: Si, P, Ca, Ti, Mn, Fe, Zn, Rb and Zr with the only sharp difference being the fact that elements Zn and Rb were only detected in the soil of continuous cropping probably due to the agrochemical applications prevalent in the study area. The inter-elemental correlation trend revealed a significant positive correlation of P to Ti and Cr (.916 and .937) at $P < 0.05$ level of significance. A similar trend was obtained for Ca to Mn and Zr (.955 and .925). There was a very strong positive significant inter elemental correlation between Ti and Cr (.994) at $P < 0.01$ level of significance. On the whole, leaving a land fallow for a relative longer period of time is beneficial in nutrients replenishment.

1. Introduction

In many parts of Africa, farmers periodically fallow their land. Fallowing is allowing land to lie undisturbed for a season or more for fertility restoration. There are so many types of fallow practices in Africa. Natural fallow for instance is simple land resting from cultivation; usually left to natural vegetation for a long period to restore soil fertility. As the length of natural fallows decrease so in general do subsequent decline in crop yields. This withdrawal of land from cultivation for a period of time to permit natural vegetation to grow on the plot is not only beneficial to nutrients replenishment but also helpful in soil textural alignment (Place et al., 2003). As long as fallow periods remain long and a low ratio of population to land area is maintained, the system is a sound soil conservation measure as the breaking of the crop cycle can lead to regeneration and the fallows can also recycle nutrients (Ruthernberg, 1971; Place et al., 2003). Improved fallow on the other hand is land resting from cultivation, but the farmer plants leguminous trees, shrubs and or herbaceous cover crops on the resting land. Improved fallow help farmers to restore the soil fertility to their land much quicker than regular fallows, so they help in cases where it is no longer possible to leave land fallow for a long period of time. Improved, planted or managed fallow practice; that is the purposeful planting of a woody or herbaceous plant to grow on a plot for a period of time is far better than fertilizer application on the soil (Place et al., 2003). The fertilizer application is the classical solution to nutrients loss especially in places where land dedicated for agriculture is scarce. Fertilizer application on soil is inherent with heavy metal pollution (Inyang, 2010). Improved fallow had been proposed as a solution to the heavy metal pollution associated with fertilizer application especially on land cultivated with vegetables and legumes (Inyang, 2010). As fallow is necessary for nutrient regeneration, compromising it will results in increased soil degradation, weed infestation and poor harvest? In addition to the benefits of natural fallows, improved fallows can achieve equal impacts of natural fallows in shorter time periods because of purposeful selection of plants, such as those that fix atmospheric nitrogen (Lal and Okigbo, 1990; Place et al., 2003) in shorter rotations. A very large area in the African and American tropics is still farmed by shifting cultivation or bush fallow techniques. The number of years of fallow tends to diminish rapidly as population pressures grow. When the fallow period becomes too short, or the cropping period too long, the soil fertility declines sharply (Nye and Greenland, 1960), and erosion becomes an increasing hazard as

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plant cover declines. This particular stress hardly occurs in Europe, because chemical fertility does not depend upon these techniques.

The ever increasing population of Nigerian over the year has resulted in a steadily increasing amount of agriculture. This population explosion poses a challenge of scarcity of land especially for agriculture. Nigeria has a limited fixed cropped land of 2813 million ha. Continuous cropping which is the case is in Nigeria has resulted in nutrients losses. It was estimated that 1,107,605 tons of nitrogen and 946,157 tons of potassium are lost from agricultural land yearly in Nigeria. Yet the scarcity of land has not encouraged the adoption of relatively longer period of fallow even when this is a very important practice (Stroorovogel and Samaling, 1990). The present investigation was conducted to actually compare the effect of relative longer yearly fallow on the concentrations of total elemental composition as compared to the soil under continuous cropping. This was very imperative as studies of this kind are scarce especially with this analytical method PIXE.

Particle induced X-ray emission (PIXE) spectroscopy is based on the interaction of charge particle (H^+) or He^{2+} at atomic level to characterize materials. PIXE is very fast and accurate method of analysis, little or no sample preparation is required. Certainly, each analytical technique commercially available have their own limitations, however, since 1993 the particle induced X-ray emission (PIXE) technique has being used successfully in agronomic applications (Cruvinel and Flocchini, 1993; Cruvinel et al., 1993; Cruvinel and Minatel, 1994). There is no literature to the best of our knowledge of the application of PIXE in the study nutrient replenishment as function duration of fallow. Moreover, in the field of soil physics, a technique which permits a non destructive, accurate and fast elemental analysis with a minimum of sample preparation effort is often desired. PIXE is an analytical technique based on the ionization of the sample atoms by the incidence of a particle beam and the subsequent emission of X-rays characteristic of the elements present in the sample (Johansson et al., 1970). The number of X-ray photons of a given element provides information on the amount of this element as part per million (ppm). It is also able to analyze very small sample quantities down to 10^{-4} g for solids and around 1 ml for liquids. In addition, it allows the simultaneous detection of practically all elements heavier than Na, a non destructive and rapid analysis. This work demonstrates the suitability of PIXE as a fast and non-destructive technique as a useful tool in measuring total elemental content in soil samples of different yearly fallow duration.

2. Materials and methods

2.1. Study area

The study area is located in Ile-Ife, Osun State in Southwest Nigeria. The study area lies approximately within longitude 4°30′E and 4°50′E and latitude 7°21′N and 7°30′N. The land use is a mixture of bush re-growth and arable crops. The study sites are underlain by Iwo soil series, which according to Symth and Montgomery (1962) developed on coarse grain rocks. Ile-Ife is humid tropics with distinct dry and wet seasons of about 4 and 8 months, respectively. The climate data put annual rainfall at 1300 mm. The mean air temperature is 13 °C. On the whole the entire study area is specifically underlain by coarse/medium grained granite geneisses (Boesse and Ocan, 1988). This terrain is unique being below a rock hill. Hence hill creep soils are expected to dominate the study area. The landscape is gently undulating terrain. The geology of the area forms a complex pattern of coarse and fine grained gneisses. The soil is derived from material of old basement complex, which is mainly made up of granite metamorphose sedimentary rock (Oyedele et al., 2008). The soil samples were collected from different identified fallow land and yearly continuous cultivated land as control. Samples were collected between November and December 2009.

2.2. Soil sampling

The soil samples used in this study were collected from different locations with specific length of fallow in Ile-Ife. As a control a continuously cropped land was also sampled. A stainless steel trowel was used to remove a core from a depth 0 to 15 cm. As a precaution, stones and plant debris were discarded. The sampling was done at random along different section in each of the chosen sites (Scott et al., 1971). In order to have a homogeneous representative sample of the soil, about 5 subsamples were taken from each fallow site and that of the control. The subsamples were placed in a clean plastic bucket and afterward homogenized. The desired sample was then removed from the bucket and the remainder discarded. The desired soils for the experiment were air-dried by placing on an expanded polystyrene tray on a metal shelving units in a ventilated warm room for few days.

2.3. Pelleting

The dried soil samples for pelleting were painstakingly ground in agate mortar/pestle. A 20% ultrapure carbon was added to each of the samples. The soil sample and the ultrapure carbon were afterward re-homogenized in the agate mortar before pelletization was done. From each sample 13 mm diameter and 1 mm thick pellets were made with Spec-caps by applying 12 tons pressure with hydraulic palletized machine. All of the above was done in the clean room to avoid contamination. Also to avoid cross contamination during grinding and pelleting, the Spec-caps, the agate mortar and pestle were cleaned by immersion for 2 h in micro-clean cleanser before and after every sample grinding and pelleting process. After immersion, the Spec-caps, the mortar, pestle and the spatula were rinsed in water filtered by million systems to remove most impurities.

2.4. Experimental equipment (the accelerator)

Figs. 1 and 2 show the layout (or schematic diagram) and photograph of CERD accelerator facility respectively used for this work. It is a model 5SDH 1.7 MV Pelletron accelerator built by the National Electrostatic Corporation (NEC), USA. This facility is equipped with a radiofrequency (RF) charge exchange ion source. The ion source is equipped to provide proton and helium ions. This equipment was installed at the Centre for Energy Research and Development (CERD) by the National Electrostatic Corporation, USA. The accelerator has provision for five beam lines and at present is maintaining one beam line that is equipped with general purpose end station for particle induced X-ray emission (PIXE), Rutherford backscattering spectroscopy (RBS), elastic recoil detection analysis (ERDA) and particle induced gamma ray emission (PIGE). The technical parameters of 5SDH Pelletron accelerator is as follows: terminal voltage 0.3–1.7 MV, proton (H^+) beam energy 0.6–3.4 MeV, $4He^+$ beam energy 0.6–3.4 MeV, $4He^{2+}$ beam energy of 0.9–5.1 MeV, proton beam current of 100–200 nA, $4He^+$ beam current 200–500 nA, and variable beam spot sizes of 1 mm, 2 mm, 4 mm, and 8 mm.

2.5. PIXE experiment

The PIXE experiments were performed using a 2.5 MeV proton obtained from CERD ion beam analysis (IBA) system. The facility is centered on a NEC 5SDH 1.7 MV Pelletron accelerator, equipped to provide proton and helium ions. The soil samples were placed on a ladder that can carry eleven 13 mm diameter samples. Soil-7

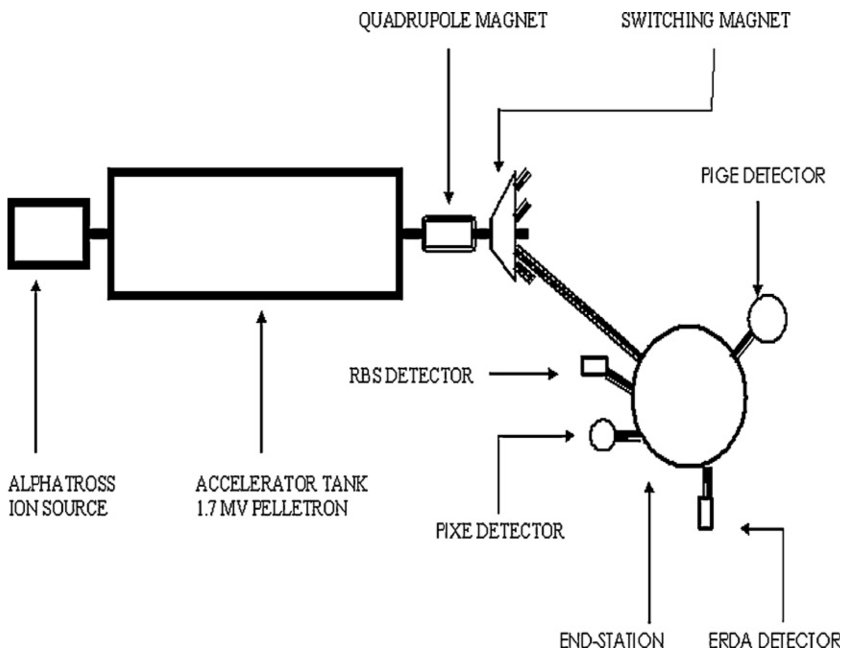

Fig. 1. Schematic layout of the accelerator facility which has found application in fallow study.

Fig. 2. General view of the IBA facility.

standard was used for the determination of the H-value which was subsequently used for analyzing the soil samples and assures the accuracy of the experimental procedure. The measurements were carried out with a beam spot of 4 mm in diameter and a low beam current of 3–6 nA. The duration of the irradiation was for about 10–20 min. A Canberra Si(Li) detector model ESLX 30-150, beryllium thickness of 25 μm, with full width half maximum (FWHM) of 150 eV at 5.9 keV, with the associated pulse processing electronics, and a Canberra Genie 2000 (3.1) MCA card interfaced to a PC were used for the X-rays data acquisition. With respect to the beam direction, the sample's normal was located at 0° and the Si(Li) detector at 45°.

3. Results and discussion

Our data suggest a linear total elemental accumulation with increasing time duration of fallow. Previous study conflict with the data presented in this study: Goldman (1998) was of the opinion that the total elemental composition of the soil is at threshold between 5 and 7 year fallow periods.

The results of the PIXE analysis obtained for soil with different duration of fallow are as presented in Table 1 while spectrum of a typical fallow soil is as presented in Fig. 3. Figs. 4 and 5 depict the

Table 1
Total elemental composition of soil sampled around land fallow as a function of yearly duration with a continuous cropped land (0 year) as a control.

	Concentration (ppm)				
	0 year	10 years	20 years	30 years	50 years
Si	276806.2 ± 30.2	153062.5 ± 389.0	16450.1 ± 368.8	14563.8 ± 56.8	183844.3 ± 351.9
P	1133.8 ± 70.8	1170.9 ± 68.9	1950.5 ± 53.17	2000.7 ± 43.8	2500.1 ± 65.4
K	29651.9 ± 36.6	2042.7 ± 15.9	2215.3 ± 14.8	2487.8 ± 21.7	7935.4 ± 68.9
Ca	1456.7 ± 439.7	609.4 ± 30.3	718.7 ± 27.2	809.6 ± 23.1	3514.7 ± 10.7
Ti	2713.0 ± 99.8	3499.9 ± 70.7	4075.3 ± 63.8	5333.7 ± 67.8	7810.6 ± 56.9
Cr	123.4 ± 8.9	293.6 ± 35.6	418.2 ± 46.9	633.8 ± 12.0	967.8 ± 16.9
Mn	443.9 ± 3.0	320.9 ± 41.7	492.6 ± 28.1	442.1 ± 32.7	987.8 ± 19.6
Fe	27552.3 ± 28.0	52553.1 ± 19.0	48861.1 ± 20.1	3444.9 ± 32.6	29082.4 ± 26.3
Zn	105.1 ± 12.7	ND	ND	ND	ND
Rb	165.6 ± 12.6	ND	ND	ND	ND
Zr	680.3 ± 8.6	346.4 ± 13.12	153.4 ± 22.5	213.6 ± 35.0	1007.7 ± 73.2

Concentrations of elements are given in ppm. ND elements below detection limit of the system. Errors are due to counting statistics.

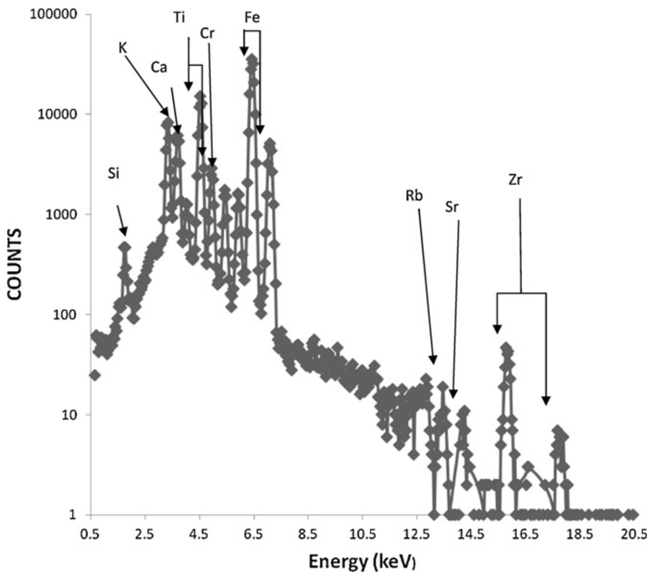

Fig. 3. PIXE spectra of a typical fallowed soil at 2.5 MeV.

Table 2
Inter-elemental correlation as a function of the different years of fallow in soil samples.

	Si	P	K	Ca	Ti	Cr	Mn	Fe	Zr
Si	1								
P	−.868	1							
K	.806	−.447	1						
Ca	−.170	.604	.223	1					
Ti	−.719	.916*	−.379	.771	1				
Cr	−.787	.937*	−.461	.698	.994**	1			
Mn	−.435	.800	.002	.955*	.878	.830	1		
Fe	.171	−.327	−.171	−.181	−.341	−.354	−.165	1	
Zr	.210	.258	.496	.925*	.504	.406	.773	−.109	1

Note: non-asterisked, not significant.
* Significant at 0.05 level.
** Significant at 0.01 level.

trend of the total elemental composition in the soils fallowed for the different yearly duration considered in this investigation. Here the objective was to study and ascertain whether leaving a land fallow for a varying number of years has any significance effect on the quantity of total elements present in the soil. The results show that the metals Si, P, Ca, Ti, Cr, Mn, Fe, Zn, Rb and Zr were detected with increasing magnitude as the year of the fallow was prolonged. The only difference being the fact that Zn and Rb were detected differently in the soil of 0 year where continuous cropping is being practiced. This could be as a result of application of agrochemicals which is associated with continuous cropping. A closer observation of the trend of other elements like P, K and Ca revealed an increase in the total elemental concentration of the soil with increasing period of land fallow. This finding is in line with the result obtained elsewhere (Ananda et al., 1991; Roder et al., 1997) but contradicts the result obtained by Goldman (1998) which is of the opinion that after the period of about 5–7 years fallowing, the total elemental composition is at threshold. Table 2 shows the inter-elemental correlation in the soil samples using the SPSS software. At 0.01 level of significance, positive significant correlation was obtained between P and Ti (r = .916) and with Cr (r = .937). Similar trend was also observed for Ca with Mn (r = .955) and Zr (r = .925) at the same level of significance. At 0.05 level, Ti also correlated positive with Cr (r = .994). Among other elements, no significant correlation was obtained whatsoever. On the whole, leaving a land fallowing for a relative longer period of time is beneficial in nutrients replenishment.

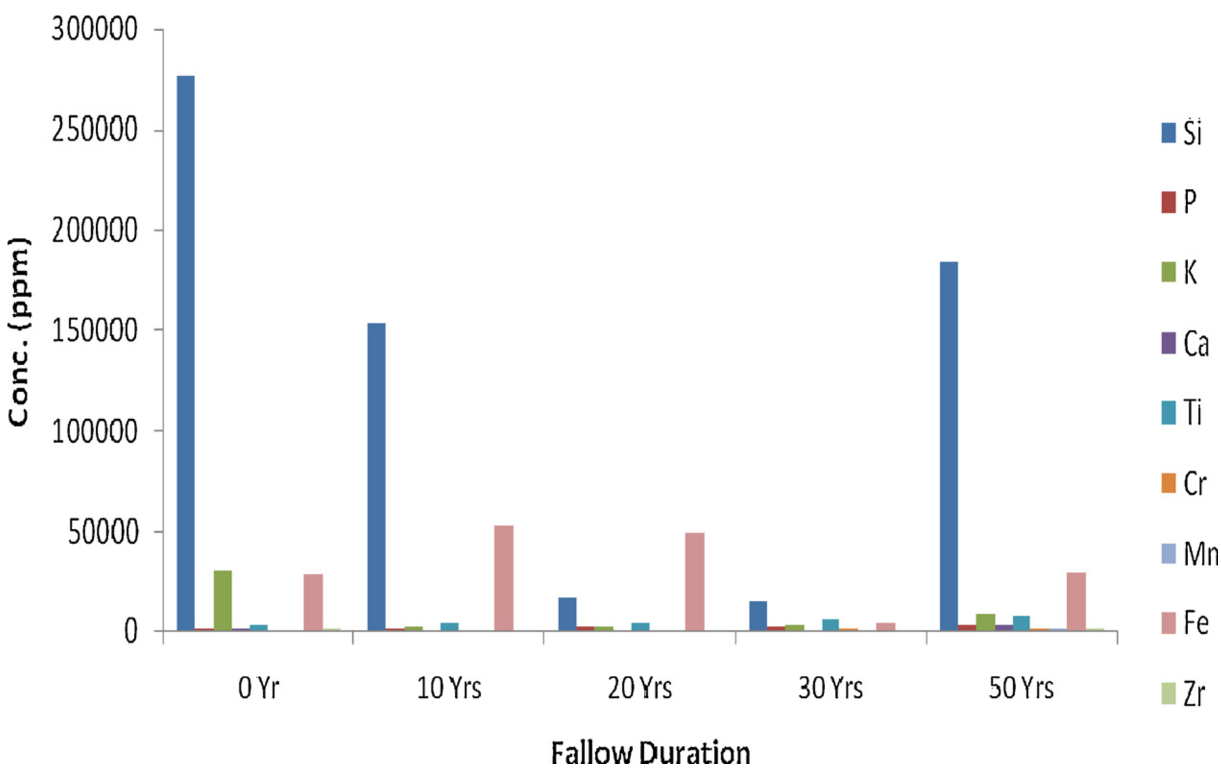

Fig. 4. Trend of total elemental composition of 9 elements in soils fallowed for different yearly durations.

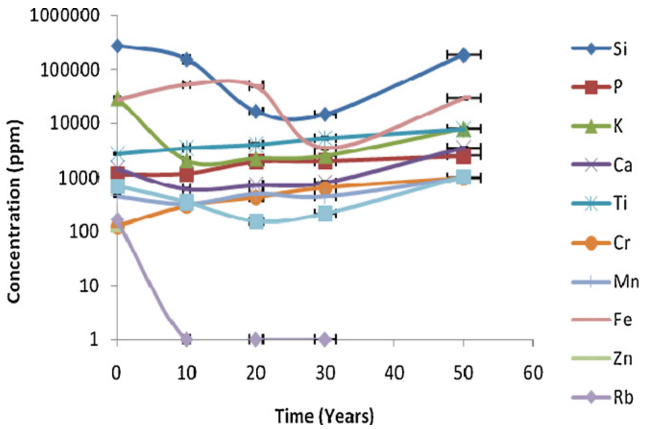

Fig. 5. Plot of concentration of soil total elements as a function of time.

4. Conclusion

Proton induced X-ray emission (PIXE) technique has been used for the first time to assess the effect of relative longer length of fallowed land with that of a continuously cropped land using a 5SDH Tandem Pelletron Accelerator available at Centre for Energy Research and Development, Obafemi Awolowo University Ile-Ife. The results at 2.5 MeV energy revealed the presence of elevated concentration of silicon, P, Ca, Ti, Mn, Fe, Zn, Rb and Zr as a function of increasing year of fallow with the only notable observation of the presence elements Zn and Rb in the soil of continuous cropping. This was probably due to the agrochemical application prevalent in the study area. The inter-elemental analysis performed using SPSS also revealed a high positive significant association among some elements such as P and Ti, P and Cr, Ca and Mn or Zr as well as Ti and Cr. This suggests that some of these elements could be used as partfinders for each other (e.g. Ca for Mn and Zr or P for Ti and Cr) in soil samples of such nature. On the whole there is no threshold to elemental elevation as a function of duration of fallowing but probably longer fallow duration should be studied longer leaving a land fallow for a relative longer period of time is beneficial in nutrients replenishment.

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