



## Original Research Article

# Assessment of Physico-Chemical Properties Influencing Mine Spoil Genesis in Chronosequence Iron Mine Overburden Spoil and Implications of Soil Quality

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## ABSTRACT

### Keywords

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Mining activities disrupts the aesthetics of landscape leading to nutritionally deprived habitats with altered ecosystem functions. Periodic monitoring of land degradation using physico-chemical indices is pre-requisite for the assessment of ecological succession to implement appropriate conservation measures. Mine spoil genesis during early years of restoration is critical, because several soil properties may require a long span of time to develop through pedogenesis to reach the soil conditions of nearby forest soil, which evaluate the degree of functional microbial processes for ecosystem recovery and used as an index of mine spoil genesis crucial for prediction of reclamation success. Physico-chemical characterization of seven age series iron mine overburden spoil revealed progressive improvement in clay % ( $r = 0.985$ ,  $p < 0.001$ ), water holding capacity, moisture content, organic C, total N and extractable P over time. Principal component analysis was able to discriminate seven different age series iron mine overburden spoil in chronosequence and nearby forest soil into eight independent clusters based on their physico-chemical properties, which showed the sign of reclamation. Further, the study suggested that the fresh iron mine overburden spoil shall take approximately 28 years to attain the soil features of nearby forest soil through the process of reclamation.

## Introduction

Soil is a dynamic system, in which continuous interactions between soil minerals, organic matter and microorganisms influences different soil physico-chemical and biological properties of terrestrial ecosystem. Soil influences vegetation development supported by microbial growth and mineralization, decomposition of organic matter (De deyn *et*

*al.*, 2004; Kardol *et al.*, 2006), and thereby undergoing pedogenesis leading to soil fertility and quality. However, land degradation of terrestrial habitats due to extensive iron mining activities often results in loss of natural ecosystem with associated biodiversity (Ezeaku and Davidson, 2008) leading to geo-environmental disasters has become a major environmental concern

(Mummey *et al.*, 2002; Xia and Cai, 2002; Lin *et al.*, 2004). The problems associated with iron mining activities are diverse that brings about inevitable natural consequences affecting environment in myriad ways causing land disturbance and change in land use pattern, disturbing natural watershed and drainage pattern, disturbing the aquifer-lowering water table, blanketing agricultural land, air and water pollution, and altering floral and faunal habitat. Besides, pit scarred landscape with huge iron mine overburden spoil usually lack biologically rich top soil, which represents disequibrated geomorphic system (Beukes *et al.*, 2008), deficient in plant nutrients (Pandey and Maiti, 2008; Sheoran *et al.*, 2008), often leads to land degradation (Singh *et al.*, 2007; Rajan *et al.*, 2010), deforestation (Bahrami *et al.*, 2010) and poses problems for revegetation (Tordoff *et al.*, 2000; Hazarika *et al.*, 2006; Pandey and Maiti, 2008), and restoration (De and Mitra, 2002; Mukhopadhyay and Maiti, 2011). Thus, iron mine overburden spoil created in the aftermath of mining activities represents rigorous conditions for both plant (Ekka and Behera, 2011; Kullu and Behera, 2011) as well as microbial community (Smejkalova *et al.*, 2003; Rath *et al.*, 2010; Maharana and Patel, 2013).

Ecological restoration and mine spoil reclamation should be dogmatic. Strategies used for restoration of iron mine spoil must address textural composition, physico-chemical characterization, soil fertility/quality, microbial community structure, soil management practices and nutrient cycling at least to the level of which existed before mining commenced. Reclamation strategies reflect a holistic approach, which not only involves the use of different reliable soil quality indicators for periodic assessment over time, but also assist the recovery of degraded ecosystem

with acceleration to continue as self-sustaining ecosystem (Yadav, 2012). Several reports suggested the slow recovery of mine spoil restoration due to constraints in microbial growth (Lindermann *et al.*, 1984; Smejkalova *et al.*, 2003; Kavamura and Esposito, 2010), and vegetation succession (Jha and Singh, 1991; Singh *et al.*, 2002; Tordoff *et al.*, 2000; Pandey and Maiti, 2008).

Short-term changes that occur in response to soil perturbation can be considered as indicators of soil quality. Soil quality is a broad concept that encompasses the physico-chemical and biological properties that sustain productivity, environmental quality, and support microbial proliferation (Doran *et al.*, 1996; Karlen *et al.*, 1997). The assessment of physico-chemical indices may be used as early and sensitive indicators of soil quality (Maharana and Patel, 2013), and ecosystem recovery (Mummey *et al.*, 2002; Xia and Cai, 2002). Therefore, monitoring periodical changes in physico-chemical properties of different age series iron mine overburden spoil over time portrays the overall success of the restoration process more accurately.

Several soil quality indicators are used to monitor changes and provide valuable information about soil management practices. However, lack of post-restoration monitoring has meant few opportunities for ecological restoration in iron mining area. Besides, reports about the physico-chemical properties and recovery of mine spoil genesis are relatively scanty. In addition, knowledge about physico-chemical characteristics of iron mine spoil is essential for guiding the conservation efforts in restoring biodiversity and site productivity. Nevertheless, the concept of adaptive management and the notion that the mine spoil genesis be regarded as a long-term

experiment is a sensible perspective. In view of the increased mining activities and decreasing soil fertility, it is of utmost concern to monitor the physico-chemical properties of different age series iron mine overburden spoil over time, which paves the way of greater understanding in the direction of improving soil quality. Keeping the above facts into consideration, the present study was designed to assess variation in physico-chemical properties in terms of textural composition (sand, silt, and clay), hydrological regimes (bulk density, water holding capacity, moisture content), chemical characteristics (pH, organic C, total N, and extractable P) in seven different age series iron mine overburden spoil in chronosequence (fresh to 25 yr) with an aim to determine mine spoil reclamation.

## **Materials and Methods**

### **Study site**

The study was carried out in Thakurani iron mining area located at Noamundi (85° 28' 02.61" east longitude and 22° 8' 33.93" north latitude), maintained by M/s. Sri Padam Kumar Jain sponge mines private limited located in the revenue district of West Singhbhum, Jharkhand, India. The study site is situated away from the mean sea level *i.e.* about 581m altitude. The area experiences a semi-arid climate, and showed the characteristics of seasonality with annual rainfall of 1340mm per year with three distinct seasons *i.e.* summer (April), rainy (July) and winter (January). Mean annual temperature and humidity is around 19.67°C and 20% respectively. Tropical dry deciduous forest is considered to be the natural vegetation of the area, but rapid development of transportation network and industrialization led to the decline of forest cover mainly due to the felling and biotic interferences. Extensive iron mining

activities led to the generation of mine spoil dumped in the form of overburden. The site is situated by a number of new, old and abandoned mines of iron overburden, which were grouped according to the time elapsed since inception such as fresh iron mine spoil (IB<sub>0</sub>), 2yr (IB<sub>2</sub>), 4yr (IB<sub>4</sub>), 6yr (IB<sub>6</sub>), 8yr (IB<sub>8</sub>), 15yr (IB<sub>15</sub>), and 25yr (IB<sub>25</sub>) respectively.

### **Spoil sampling**

Sampling was done in accordance with general microbiological protocol from seven different age series iron mine overburden (IB<sub>0</sub>, IB<sub>2</sub>, IB<sub>4</sub>, IB<sub>6</sub>, IB<sub>8</sub>, IB<sub>15</sub> and IB<sub>25</sub>) within a peripheral distance of 10 km from the core iron mining area. Besides, the nearby native forest soil (NF) was selected adjacent to the core coal mining area for comparison. During sampling, each site was divided into 3 blocks, and five mine overburden spoil samples were collected randomly from 0-15cm soil depth by digging pits of (15 x 15 x 15) cm<sup>3</sup> size. The samples collected from each block were referred as 'sub-samples', and were thoroughly mixed to form one 'composite sample' obtained from each overburden. Similar strategy of sampling has been followed to obtain three composite samples from each site in three different seasons (summer, rainy, and winter). The samples were subjected to sieving (0.2 mm mesh size) and stored at 4°C until analyzed.

### **Spoil texture**

Spoil texture analysis included the estimation of clay (< 0.002 mm), silt (0.05 mm - 0.002 mm) and sand (2 mm - 0.06 mm) %. Spoil sample (50g) was taken in a heat resistant bottle calibrated upto 250ml by adding 125ml of water, and the mixture was swirled to wet the spoil thoroughly. Then, 20ml of 30% NaOH was added and the bottle was gently rotated. Few drops of

amyl alcohol was added to the mixture and kept in boiling water bath. Thereafter, 2g of sodium hexametaphosphate was added, the volume was made up to 250ml by adding water, and was subjected to shaking for 28 hr. The contents were transferred to sedimentation cylinder and the volume was made up to 1 liter. A blank was maintained by dissolving 2g of sodium hexametaphosphate in water. Both experimental and blank samples were incubated in water bath maintained at  $(25 \pm 2)^{\circ}\text{C}$ . After 30 min, the sample was mixed vigorously, and the Bouyoucos hydrometer readings were taken at 40sec and 5hr for the samples, and at 5hr for the blank. The temperature of the water bath was recorded. The percentage of textural composition (sand, silt and clay) was determined as per calculations.

40 sec (corr) = 2 (40 sec reading – 40 sec blank + T)

5 hr (corr) = 2 (5 hr reading – 5 hr blank + T)

Where, T = Temperature corrections:

For every degree centigrade above  $20^{\circ}\text{C}$  (d),  
 $T = 0.3 \times d$

For every degree centigrade below  $20^{\circ}\text{C}$  (d),  
 $T = - 0.3 \times d$

% sand =  $100 - 40 \text{ sec (corr)}$

% silt =  $40 \text{ sec (corr)} - 5\text{hr (corr)}$

% clay =  $5 \text{ hr (corr)}$

### **Bulk density**

Bulk density was calculated following the method prescribed in TSBF Handbook (Anderson and Ingram, 1992). A pit of  $(10 \times 10 \times 20) \text{ cm}^3$  was dug and the soil was excavated. The collected soil was dried in an oven at  $105^{\circ}\text{C}$  for 24 hr, and then the dry weight was determined. The pit was filled with known volume of dry sand. The bulk density ( $\text{g}/\text{cm}^3$ ) was calculated as: [weight of excavated soil (in g)/ vol. of sand ( $\text{cm}^3$ )].

### **Water holding capacity (WHC)**

Water holding capacity was determined following the protocol proposed by Mishra (1968). Mine spoil samples from the different age series iron mine overburden were collected, air dried and sieved (0.5 mm mesh). A brass box with perforated bottom was taken and a Whatman filter paper (No. 42) was placed on the perforated bottom. A split brass ring was used to press the filter paper to its position. The box along with the filter paper was weighed and recorded as ( $W_1$ ). The brass box was filled with soil with constant tapping to ensure uniform packing. The soil packed box was placed on a petridish and water was added to maintain the depth of 1 cm. From time to time, water was added to maintain the depth. The box was left in the position for 12 hr. After that, the box was removed, subjected to surface drying with blotting paper and the weight of the box was recorded ( $W_2$ ). The box was then placed in the oven at  $105^{\circ}\text{C}$  for 24 hr and the dry weight was recorded as ( $W_3$ ). Water absorbed by the filter paper was determined by saturating five filter papers with water and weighing after rolling with a glass rod. The average amount of water absorbed by one filter paper was determined as ( $W_4$ ). The water holding capacity (%) of different iron mine overburden spoil was calculated as:  $[(W_2 - W_3 - W_4)/(W_3 - W_1)] \times 100$ .

### **Moisture content**

About 10g of iron mine spoil sample was taken ( $W_1$ ). The sample was oven dried at  $105^{\circ}\text{C}$  for 24hr or more till a constant dry weight was obtained ( $W_2$ ). The soil moisture (%) in different age series iron mine overburden spoil was calculated as:  $[(W_1 - W_2)/10] \times 100$ .

### **Soil pH**

Air dried soil of 20g was taken in a beaker and 50ml water was added. The mixture (1:2.5 ratio of soil:water) was stirred for 10 min and was allowed to stand for 30 min. The pH was measured by using the electronic digital pH meter (Make: Systronics, Model: MK VI).

### **Organic carbon**

Organic carbon (OC) content was determined using titration method of Walkley and Black (Mishra 1968). To 5g of oven dried mine spoil were weighed into 500ml Erlenmeyer flask. Each of the samples beakers were subjected to rapid dichromate oxidation (10ml of 1N  $K_2Cr_2O_7$ ) by addition of 20ml of conc.  $H_2SO_4$ . The resulting suspension was swirled (5 min) and allowed to stand for 30 min to reduce the heat generated by exothermic reaction. The suspension was diluted with 200ml of distilled water followed by 1ml of 85%  $H_3PO_4$  to eliminate interference from iron III ( $Fe^{3+}$ ) ion that may be present and 1ml of diphenylamine indicator. The mixture was titrated against 1N  $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$  until the colour of the mixture flashed to green. Then, 0.5ml of 1N  $K_2Cr_2O_7$  was added, and titration was completed by adding 1N  $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$  till the last traces of blue colour disappeared. Soil organic carbon (%) was calculated as:  $[(V_1 - V_2)/W] \times 0.003 \times 100$ ; where,  $V_1$  = vol. of 1N  $K_2Cr_2O_7$ ;  $V_2$  = vol. of 1N  $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ ; W = wt. of sample (g).

### **Total Nitrogen**

Total nitrogen (TN) content was determined by Kjeldahl method (Jackson, 1958). Spoil sample of 10g was taken in 300ml Kjeldahl flask, and moistened with 25ml of distilled water. After 30 min, 20g of  $Na_2SO_4$  and the

catalyst mixture (20g  $CuSO_4$ , 3g of  $HgO$ , 1g selenium powder) was added. To one part of mixture, 20 parts of anhydrous  $Na_2SO_4$  and a pinch of granulated zinc were added followed by 35ml of conc.  $H_2SO_4$ . The suspension was subjected to low heat treatment for 30 min till the digest become yellow and colourless followed by cooling by addition of 100ml of water, and allowed to stand for 5 min. The supernatant was then transferred into a 500ml conical flask and 25ml of 4% boric acid and 5 drops of mixed indicator (0.5g bromocresol green and 0.1g methyl red dissolved in 100ml of 95% ethyl alcohol) was added. The glass tube attached to lower end of condenser was dipped into boric acid solution. The condenser was connected to the flask and 100ml of 40%  $NaOH$  was added slowly through separating funnel. By heating the mixture, 150ml of distillate was collected in a conical flask and was titrated against N/14  $H_2SO_4$  till faint pink colour was reached. A blank was run instead of spoil. Total nitrogen (%) was calculated as:  $[(T - B) \times N \times 14.007 \times 100]/W$ ; where, T = vol. of titrant (in ml) used against sample, B = vol. of titrant (in ml) used against blank, N = normality of titrant, and W = wt. of sample (g).

### **$NaHCO_3$ extractable Phosphorous**

The extractable phosphorous (EP) content was estimated using chlorostannous reduced molybdophosphoric blue colour method in HCl (Olsen and Sommers, 1982). The molybdophosphoric acid is thought to be formed by the substitution of oxygen of molybdate ions with phosphorous as the central coordinating atom. Sieved and air dried spoil sample of 5g was transferred to a 250ml conical flask and to it 50ml of 0.03N  $NH_4F$  in 0.025N HCl was added and was shaken for 5 min and filtered immediately. To 2.5ml of filtrate, 7.5ml of ammonium molybdate was mixed thoroughly followed

by the addition of freshly prepared 0.5ml of stannous chloride and allowed to stand for 4-20 min. Absorbance was taken at 660 nm, and the extractable P content was expressed in  $\mu\text{g/g}$  spoil.

### Statistical analysis

Statistical analyses were performed using Microsoft Excel 97. The simple correlation analysis between different soil properties was performed using SPSS software. Further, principal components analysis (PCA) was performed in order to discriminate different age series iron mine overburden spoil and nearby forest soil based on the physico-chemical indices using Statistrix PC DOS Version-2.0 (NH Analytical software).

### Results and Discussion

The textural composition, bulk density, different hydrological regimes and pH of different age series iron mine overburden (IB<sub>0</sub>, IB<sub>2</sub>, IB<sub>4</sub>, IB<sub>6</sub>, IB<sub>8</sub>, IB<sub>15</sub>, and IB<sub>25</sub>) as well as nearby forest soil (NF) have been represented (Table 1). The textural analysis indicated an increasing trend in clay percentage from IB<sub>0</sub> (4.4%) to IB<sub>25</sub> (11.2%). Similar trend was also observed in case of silt percentage, which ranged from 7.8% (IB<sub>0</sub>) to 13.5% (IB<sub>25</sub>). However, the decline trend in sand percentage was evident from the study that varied from 87.8% (IB<sub>0</sub>) to 75.3% (IB<sub>25</sub>). The study indicated relatively higher clay percentage in nearby NF soil (13.3%) as compared to different age series iron mine overburden spoil across the sites. Gradual establishment of vegetation cover may be the reason for the increase in clay fraction (Jha and Singh, 1991; Banerjee *et al.*, 2000; Dutta and Agrawal, 2002), which is a sustainable approach to stabilize and reclaim the disturbed land in mining area (Tordoff *et al.*, 2000) by preventing soil

erosion (Conesa *et al.*, 2007b) and enhancing microbial mineralization (Li, 2006; Conesa *et al.*, 2007a; Mendez and Maier, 2008). Besides, the analysis of variance (ANOVA) indicated that the consistent increase in clay percentage in different age series iron mine overburden spoil was found to be significant ( $p < 0.001$ ).

Soil texture affects other soil properties, which in turn determine microbial growth and activity, and hence reported as key determinants of microbial ecology. Increasing clay fraction leads to restoration of topsoil profile, which has been the conventional approach in restoration of abandoned mining area (Bradshaw and Huttl, 2001; Prach and Pysek, 2001; Zhao *et al.*, 2009; Alday *et al.*, 2011; Kullu and Behera, 2011), as because it being a source and sink of nutrients and organic content promoting micro-aggregation (Schafer, 1984; Gupta and Germida, 1988), soil structural stability and nutrient retention capacity (Garcia *et al.*, 1996). Further, the clay fraction acts as buffer negating the adversities of the derelict mine land and ameliorating soil physico-chemical properties and different hydrological regimes in favour of vegetation development (Yan *et al.*, 2013).

The textural composition and particle size distribution influences different hydrological regimes including bulk density, water holding capacity and moisture content. Bulk density can be considered as an indicator of problems indicating the disturbed texture of mine spoil. Bulk density exhibited a decline trend from IB<sub>0</sub> (1.852  $\text{g/cm}^3$ ) to IB<sub>25</sub> (1.332  $\text{g/cm}^3$ ) with the increase in age of mine overburden (Table 1). Higher bulk density in IB<sub>0</sub> may be due to the removal of vegetation arising from the mining activities (Ezeaku and Ikemefuna, 2012). Fresh mines soil

poses limited plant growth, as they are unable to extend roots effectively through high bulk density mine soils (Shrooran *et al.*, 2010). The IB<sub>25</sub> with reduced bulk density exhibited closer resemblance with nearby NF soil (1.259 g/cm<sup>3</sup>), which may be due to the gradual accumulation of clay fraction and organic matter input in IB<sub>25</sub> supported by gradual establishment of vegetation promoting macro-aggregation (Gupta and Germida, 1988; Ohta and Effendi, 1992; Sadhu *et al.*, 2012). Importance of bulk density lies with the fact that it regulates the space, air and water availability to soil microbes (Foissner, 1992). The decline in bulk density can be interpreted as a reduction in soil compactness because of the development of soil micropore space (Ohta and Effendi, 1992). Clay fraction has the ultimate bearing of soil bulk density, and hence increased level of clay fraction contributes to the development of soil micropore space that reduces soil bulk density. In light of the concept, the different age series iron mine overburden spoil have higher bulk density and low porosity as compared to nearby NF soil. Besides, bulk density could be affected by land use and soil types (Han *et al.*, 2010). Analysis of variance (ANOVA) indicated negative correlation of bulk density with the age of mine spoil ( $r = 0.980$ ,  $p < 0.001$ ), which indicated that 96.09% variability in bulk density was accounted by the age of iron mine overburden.

The water holding capacity showed a reverse trend, which varies from 24.501% (IB<sub>0</sub>) to 44.509% (IB<sub>25</sub>). The moisture content also showed the similar trend *i.e.* minimum in IB<sub>0</sub> (6.643%) and maximum in IB<sub>25</sub> (10.886%), which may be due to the positive influence of canopy cover in IB<sub>25</sub> that prevented the loss of water through evaporation by not allowing direct exposure of soil surface to incoming radiation (Bird *et*

*al.*, 1984). The WHC and MC content in nearby NF soil were found to be 46.648% and 11.329% respectively (Table 1). Soil texture and organic matter content are the key components for the assessment of water holding capacity (Vengadaramana and Jashothan, 2012). Across the sites, higher WHC and MC in nearby NF soil as compared to different age series iron mine overburden spoil may be attributed to dense vegetation cover and gradual supplements of organic matter (Sigh *et al.*, 2004), increased aggregation and total pore space (Vengadaramana and Jashothan, 2012).

Soil reaction is often modelled as a positive linear relationship with soil fertility and productivity, where the soil with higher pH indicates a better soil. Soil pH should be site-specific, and could be added as additional criterion for soil classification and mapping used for soil quality assessment (Sheoran *et al.*, 2010). The pH of different age series iron mine spoil was estimated to be in acidic range, which ranges from 6.14 (IB<sub>0</sub>) to 6.77 (IB<sub>25</sub>) across the sites (Table 1). However, the pH of nearby NF soil was found to be 6.83. The acidification of mine spoil may be due to different minerals deposition (Jha and Singh, 1991; Suzuki *et al.*, 1999; Dutta and Agrawal, 2002), accumulation of organic C and formation of organic acids over time (Sourkova *et al.*, 2005), or due to oxidation of residual elements such as iron and sulphur (Hazarika *et al.*, 2006) that hinder the release of available essential plant nutrients (Rai *et al.*, 2011). The data indicated gradual improvement in pH in different age series iron mine overburden spoil, which may be due to both passive and active reclamation process either by natural succession or by plantation/vegetation development, available micronutrients, and shift in microbial community associated with mycorrhiza over time (Jha and Singh, 1991; Dutta and

Agrawal, 2002). Promotion of organic matter decomposition on derelict mined soil also has been reported to lower soil acidification (Sahani and Behera, 2001). Improving soil chemical condition by the reduction of soil acidity has been well explained (Johnson and Skousen, 1995; Suzuki *et al.*, 1999). Hence, soil pH can be used for the assessment of soil quality in terrestrial environment (Rai *et al.*, 2011).

The study indicated wide variation in organic C, which varied from 0.142% to 2.228% with minimum in IB<sub>0</sub> and maximum in IB<sub>25</sub> (Table 2). However, higher level of OC was recorded in nearby NF soil (2.469%) as compared to different age series iron mine spoil. The gradual increase in OC from IB<sub>0</sub> to IB<sub>25</sub> was found to be positively correlated with the age of mine spoil ( $r = 0.912$ ;  $p < 0.001$ ). The study indicated gradual improvement in OC from a nutrient deficient iron mine overburden spoil to an enriched NF soil over time, which may be due to the establishment of vegetation (Fu *et al.*, 2010; Wang *et al.*, 2011), input of litter from vegetation compartment and its decomposition during the course of passive or active restoration (Maiti and Ghose, 2005). Organic matter acts as biological "glue" that cements individual soil particles together into aggregates and making soil resistance to erosion (Shen *et al.*, 1984). Positive correlation was observed between clay percentage and OC content between different age series iron mine overburden spoil ( $r = 0.963$ ,  $p < 0.01$ ) (Table 3). Increase in OC content exhibited positive correlation with the increase in clay fraction in ecologically derelict mining land areas was substantiated by several workers (Roberts *et al.*, 1981; Marrs *et al.*, 1981; Maharana and Patel, 2013). Soils with higher clay percentage can store more organic C (Muller and Hoper, 2004). Small difference in clay percentage may result in

significant differences in organic C accumulation in reclaimed mine soil (Chodak and Niklinska, 2012). Besides, the variation in organic C depends upon land use, soil type and terrain (Han *et al.*, 2010), which have great impact on soil nutrient status (Wong, 2003). Further, the organic C in association with primary soil particles is reported to promote soil aggregation, structural stability and nutrient retention capacity (Garcia *et al.*, 1996), and hence considered to be the reliable indicator for monitoring soil quality and land degradation (Rajan *et al.*, 2010).

Similarly, the total N and extractable P also showed gradual improvement in different age series iron mine overburden spoil over time. The TN ranged from 0.004% (IB<sub>0</sub>) to 0.187% (IB<sub>25</sub>). Similarly, the EP varies from 70.445  $\mu\text{g P/g}$  soil (IB<sub>0</sub>) to 945.678  $\mu\text{g P/g}$  soil (IB<sub>25</sub>) across the sites (Table 2). However, the TN and EP content in nearby NF soil were found to be 0.245% and 1091  $\mu\text{g P/g}$  soil respectively. The lower amount of EP in IB<sub>0</sub> may be due to slightly acidic nature of fresh mine spoil, which restricted microbial mineralization and organic decomposition (Rai *et al.*, 2011). Significant variation in TN ( $r = 0.891$ ;  $p < 0.05$ ) and EP ( $r = 0.820$ ;  $p < 0.001$ ) in different age series iron mine spoil with respect to the age of mine overburden was revealed by simple correlation analysis. Gradual accumulation of soil nutrients from IB<sub>0</sub> to enriched NF soil may be attributed to the input from plant species capable of nitrogen fixing potential as well as development of mycorrhiza and other nutrient immobilizing microbial colonization. The total N and extractable P are found to be deficient in mine overburden spoil (Sheoran *et al.*, 2008), and has been identified to contribute soil fertility in both managed and natural ecosystems (Kucharik *et al.*, 2001).



Nevertheless, the variation in OC content with respect to different mine overburden spoil was positively correlated with TN ( $r = 0.991$ ;  $p < 0.01$ ) and EP ( $r = 0.982$ ;  $p < 0.01$ ) across the sites (Table 3). The study clearly indicated that the iron mine overburden spoil showed the sign of restoration due to the gradual accumulation of OC, TN, and EP over time.

Further, principal component analysis was performed in order to discriminate seven different age series iron mine overburden spoil (IB<sub>0</sub> → IB<sub>25</sub>) as well as nearby NF soil. The analysis can able to segregate eight different soil profiles into independent

cluster based on their physico-chemical properties, in which the Z<sub>1</sub> and Z<sub>2</sub> components accounts for 99% cumulative variance (Figure 1).

The mining activities limit the establishment and growth of native plant species resulted in the loss of forest cover around the iron mining area, previously which was under the dense deciduous forest. The study revealed gradual improvement in different physico-chemical properties in chronosequence iron mine overburden spoil over time, which indicated the pace and progress of reclamation.

**Table.1** Textural composition, bulk density, water holding capacity, moisture content and pH of mine spoil samples collected from seven age series iron mine overburden (IB<sub>0</sub> → IB<sub>25</sub>) as well as nearby forest (NF) soil. Values expressed in mean ± SD; n = 3

Textural composition	Different age series iron mine overburden spoil							Forest soil (NF)
	IB0	IB2	IB4	IB6	IB8	IB15	IB25	
Sand (%)	87.8 ± 2.15	85.9 ± 1.84	84.8 ± 1.75	83.4 ± 1.89	81.5 ± 1.64	79.7 ± 1.55	75.3 ± 1.36	72.5 ± 2.05
Slit (%)	7.8 ± 0.45	8.4 ± 0.33	9.1 ± 0.41	9.9 ± 0.29	10.9 ± 0.37	11.8 ± 0.41	13.5 ± 0.52	14.2 ± 0.66
Clay (%)	4.4 ± 0.33	5.7 ± 0.21	6.1 ± 0.28	6.7 ± 0.34	7.6 ± 0.38	8.5 ± 0.29	11.2 ± 0.24	13.3 ± 0.31
Bulk density (g/cm <sup>3</sup> )	1.852 ± 0.036	1.794 ± 0.029	1.715 ± 0.035	1.664 ± 0.028	1.593 ± 0.034	1.405 ± 0.033	1.332 ± 0.029	1.259 ± 0.021
WHC (%)	24.501 ± 1.235	26.422 ± 1.558	28.067 ± 2.013	32.311 ± 2.152	37.457 ± 1.942	40.338 ± 1.675	44.509 ± 2.045	46.648 ± 2.164
Moisture (%)	6.643 ± 0.206	6.985 ± 0.211	7.106 ± 0.198	7.422 ± 0.201	8.391 ± 0.168	9.915 ± 0.176	10.886 ± 0.155	11.329 ± 0.198
Soil pH	6.14 ± 0.08	6.24 ± 0.06	6.36 ± 0.05	6.49 ± 0.06	6.59 ± 0.05	6.62 ± 0.06	6.77 ± 0.08	6.83 ± 0.08

**Table.2** Organic C, total N, and extractable P content in mine spoil samples collected from seven different age series iron mine overburdens (IB<sub>0</sub> → IB<sub>25</sub>) as well as nearby forest (NF) soil. Values expressed in mean ± SD; n = 3

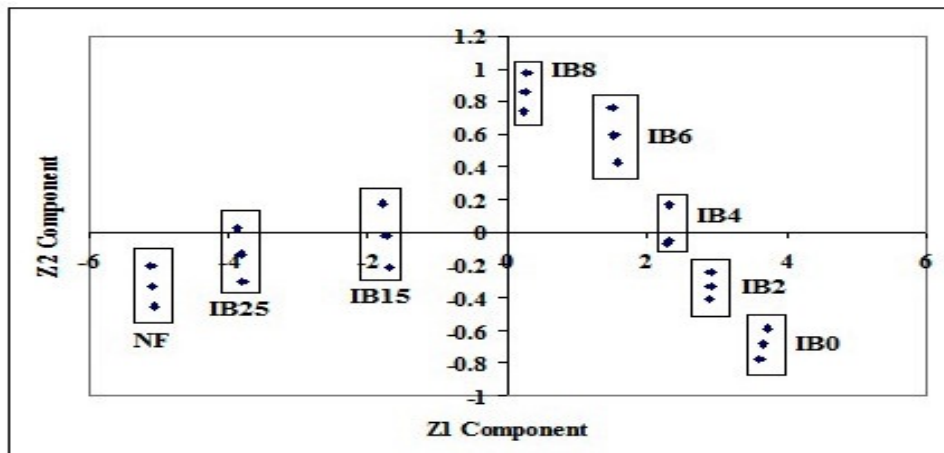
Textural composition	Different age series iron mine overburden spoil							Forest soil (NF)
	IB0	IB2	IB4	IB6	IB8	IB15	IB25	
Organic C (%)	0.142± 0.029	0.218± 0.024	0.284± 0.028	0.355± 0.034	0.815 ± 0.039	1.648 ± 0.041	2.228 ± 0.045	2.469 ± 0.052
Total N (%)	0.004± 0.001	0.007± 0.002	0.011± 0.004	0.015± 0.003	0.053 ± 0.002	0.125 ± 0.005	0.187 ± 0.007	0.245 ± 0.008
Extractable P (µg P/g soil)	70.445 ±2.304	76.836 ±3.442	84.552 ±2.987	91.707 ±3.416	112.542 ± 8.588	645.817 ±11.508	945.678 ±15.647	1091.509 ± 25.551

**Table.3** Simple correlation coefficients (*r*) between different soil properties

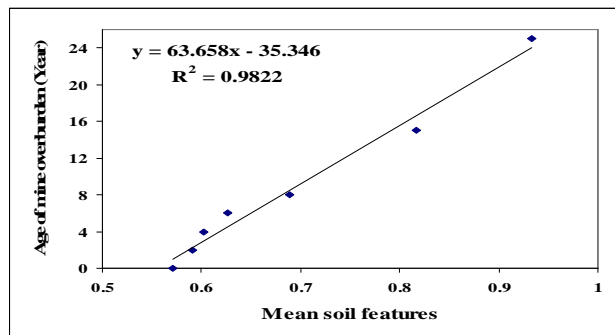
Parameters	Sand	Slit	Clay	BD	WHC	MC	pH	OC	TN	EP
Sand	1									
Slit	-0.994**	1								
Clay	-0.996**	0.979**	1							
BD	0.979**	-0.990**	-0.962**	1						
WHC	-0.975**	0.992**	0.953**	-0.986**	1					
MC	-0.977**	0.983**	0.964**	-0.989**	0.976**	1				
pH	-0.962**	0.978**	0.940**	-0.965**	0.980**	0.931**	1			
OC	-0.972**	0.972**	0.963**	-0.978**	0.959**	0.997**	0.908**	1		
TN	-0.973**	0.960**	0.975**	-0.959**	0.936**	0.982**	0.885**	0.991**	1	
EP	-0.942**	0.929**	0.944**	-0.940**	0.897**	0.968**	0.839**	0.982**	0.987**	1

\*\* Correlation is significant  $p < 0.01$ ; and \* correlation is significant  $p < 0.05$ .

**Figure.1** Principal component analysis based on physico-chemical properties in seven different mine overburden spoil (IB<sub>0</sub> → IB<sub>25</sub>) as well as nearby NF soil



**Figure.2** Relationship between mean soil features with different age series iron mine overburden spoil in chronosequence



Considering the tropical dry deciduous forest as natural vegetation of the study site, an attempt was made to compare the soil features of different age series iron mine overburden spoil in chronosequence (IB<sub>0</sub> → IB<sub>25</sub>) over a period of 25 years with nearby forest (NF) soil. For the purpose, the proportionate level of these parameters for different age series iron mine overburden spoil were calculated by taking the soil parameters of nearby NF soil as unit. Further, attempt was made to calculate the time period required for restoration of IB<sub>0</sub> to reach the nearby NF soil condition. Accordingly, a positive correlation was observed ( $r = 0.991$ ;  $p < 0.001$ ) between the mean soil feature and age of mine overburden, which explained 98.22% variability in soil physico-chemical properties with respect to the age of mine overburden spoil (Figure 2). Taking the value of nearby forest soil data (*i.e.* 1) as 'X', the equation was used to calculate the age of NF soil *i.e.* 28.312 years.

In view of the foregoing, the objective of the present investigation is thus stocktaking of the variation in different physico-chemical indices in seven different age series iron mine overburden spoil, which are ecologically hostile may prove useful in formulating reclamation strategies and eventual eco-restoration. Besides, the

objective of ecological rehabilitation is to accelerate natural succession in order to increase biological productivity, soil quality and biotic control over biogeochemical fluxes within the recovering ecosystems. The present investigation was performed to evaluate the variation in different physico-chemical properties in seven different age series iron mine overburden spoil and nearby forest soil. The study suggested that the soil quality is intimately related to physico-chemical properties, and hence their evaluation will facilitate to characterize soil fertility status and productivity. The physico-chemical indices appeared to be more informative and could therefore be used for the selection of appropriate reclamation strategies. Significant correlation between the clay fraction and organic C in different age series iron mine overburden spoil indicated that they are the most undervalued component for mine spoil reclamation. Based on the comparative assessment of physico-chemical properties in seven age series iron mine spoil and nearby forest soil into account, the study suggested that the fresh iron mine overburden spoil (IB<sub>0</sub>) to attain the soil feature of the nearby forest soil through the process of reclamation shall take approximately 28.312 years provided the mine spoil habitat is not subjected to any other interferences or degradation *etc.*

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