

Development of an x-ray method for mineralogical analysis of Africa soils using a benchtop diffractometer

Mercy Nyambura^{1*,2}, Michael Gatari¹, Stephen Hillier^{3*}, Keith D. Shepherd², Esala Martti⁴ and Benson Mochoge⁴

¹Institute of Nuclear Science, College of Architecture and Engineering, University of Nairobi, Nairobi, Kenya, ²World Agroforestry Centre (ICRAF), United Nations, Nairobi, Kenya, ³The James Hutton Institute, Aberdeen, Scotland, ⁴MTT Agrifood Research Finland, ⁵Kenyatta University, Nairobi, Kenya.

Introduction

Soil mineralogy is a principal determinant of many soil functional properties.

Africa soil mineralogy has not been widely researched, especially as a predictive tool.

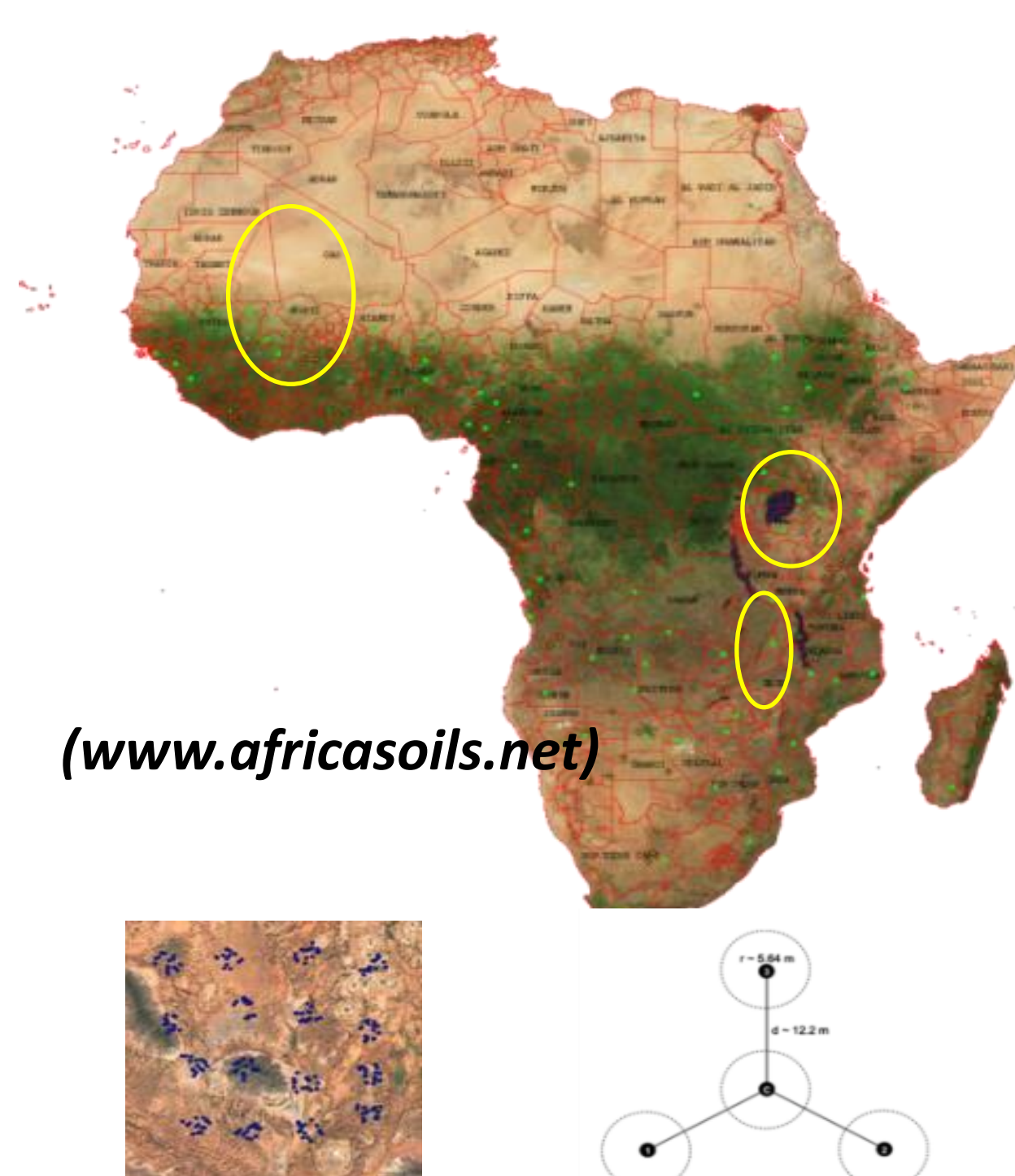
Recent developments in instrument designs, and capabilities including the launch of bench-top X-ray Diffractometers (XRD) have widened the possible application areas for high-throughput X-ray diffraction (XRD) as a powerful complementary tool for soil screening.

This study was conducted to develop a protocol for high precision and rapid throughput mineralogical analysis of Africa soils using a benchtop diffractometer

Methods

Efficient and reproducible procedures capable of high precision XRD analysis were developed and used.

Sampling approach:



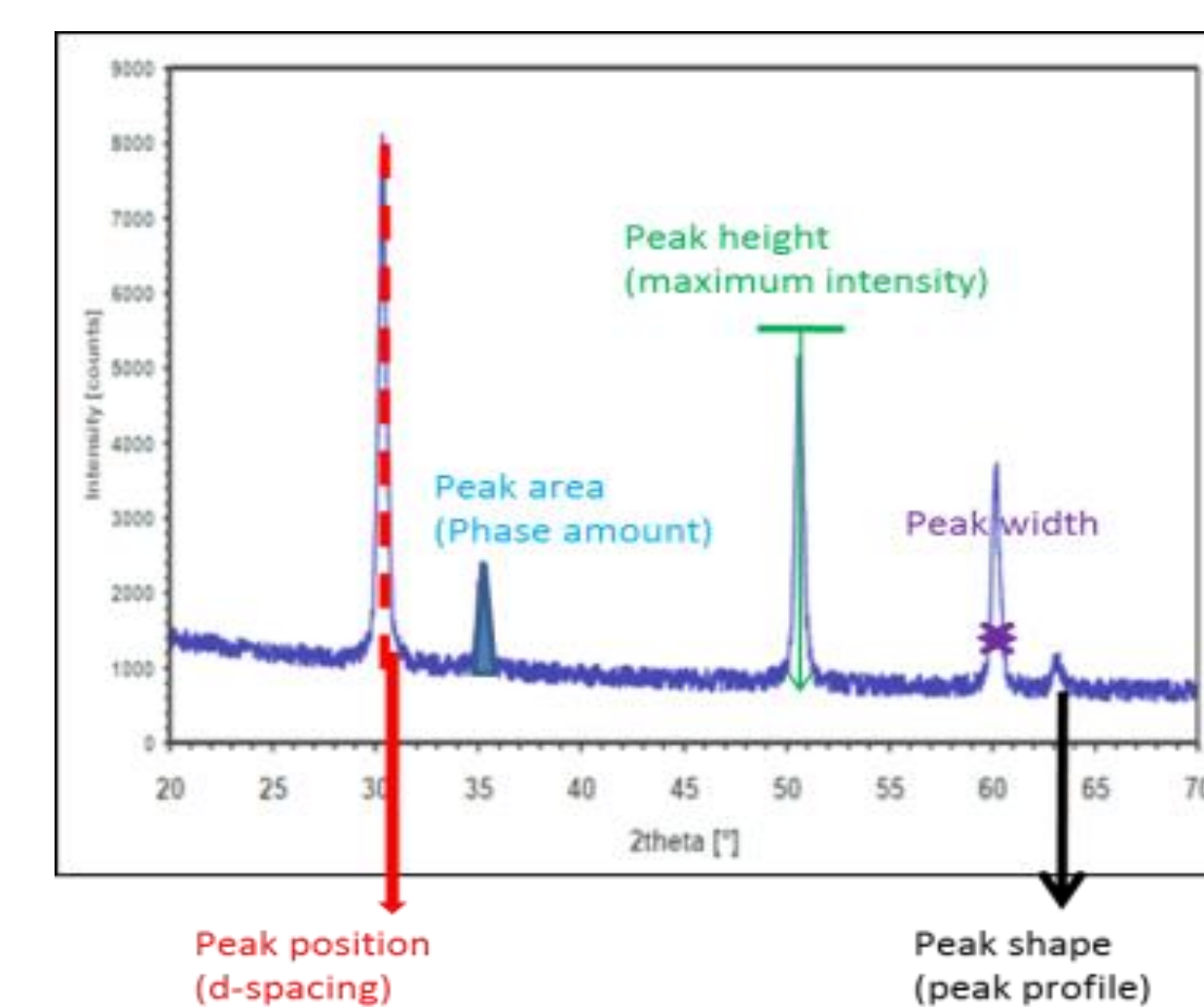
Sample Preparation:



Goals:

- Random powder
- Reduce preferred orientation
- Homogeneity
- Reduce specimen error
- Particle statistics

XRD Measurement:



Good Signal, low background and low noise

Results

Diffraction patterns of a multiphase sample powder mounts for 6 different loadings by 2 different operators and using 3 methods showed:

- Inconsistent between phases from the portion prepared by conventional fine grinding Fig 1a, and in contrast excellent repeatability by, and between, all two operators between phases prepared by McCrone milling in ethanol Fig 1b. These results were also comparable to phases prepared by spray drying.
- High quality diffractograms for the 160 samples (from 10 sites) identified and semi quantified Fig 1c. These demonstrate the effects of adequate sample preparation procedures, reduced preferred orientation, reproducibility, visual data comparison and matching unknowns with known patterns from the PDF files

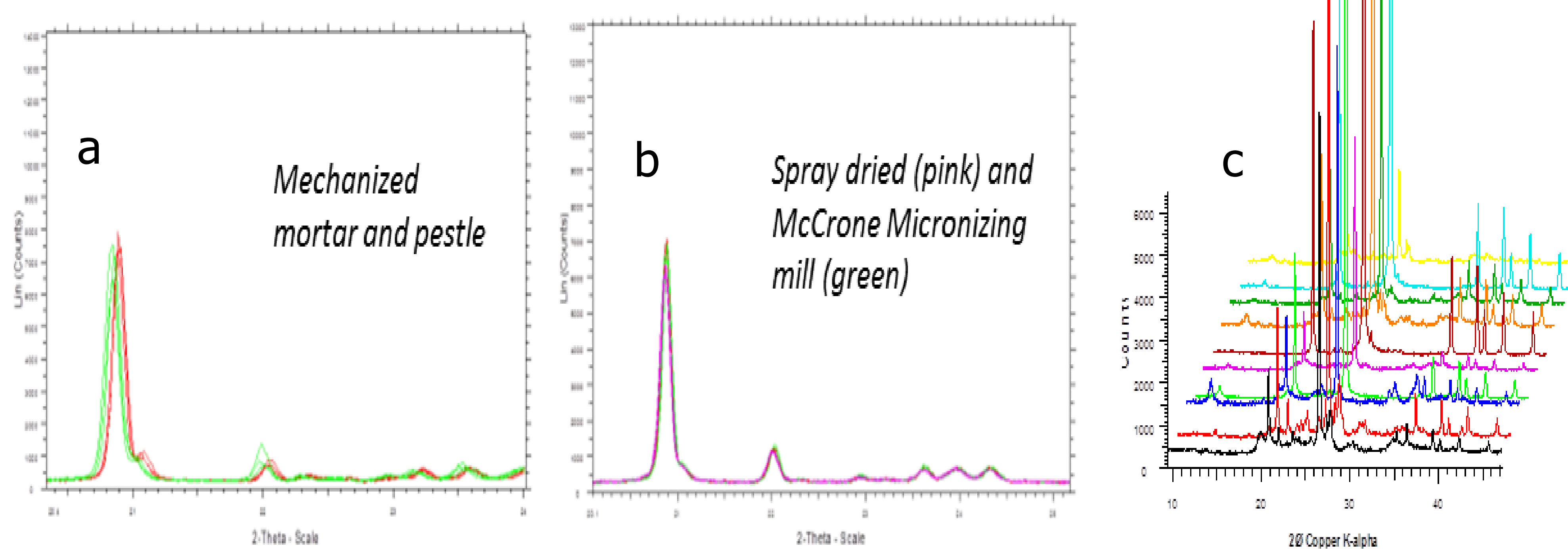


Fig. 1 Diffraction patterns of a multiphase sample powder mounts from 6 different loadings by 2 different operators. Powders prepared by a) conventional milling in a mechanized mortar and pestle. b) McCrone Micronizing mill (green, red,) Spray-dried (pink). c) Diffraction patterns of samples from the ten AfSIS sites

Conclusion

The study has presented an accurate, low cost and throughput protocol for XRD analysis, suitable for routine application by soil testing laboratories, and a way to characterize Africa soil mineralogy quickly and at scale