DEVELOPING MATRIX-MATCHED EMPIRICAL CALIBRATIONS FOR EDXRF ANALYSIS OF PEAT-ALTERNATIVE GROWTH MEDIA

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INTRODUCTION

- Growing media regulate water, nutrients, aeration, and root support in soilless systems requiring precise control.
- The shift from **peat** to **sustainable alternatives** introduces high variability in media properties.
- Reliable characterisation is essential to prevent **nutrient imbalances** and **contamination risks**.
- **Spectroscopic techniques (XRF, IR)** offer rapid, non-destructive analysis, supporting practical applications in **ornamental and food crop horticulture**.

Objectives

Develop and validate benchtop Energy Dispersive X-ray Fluorescence methods

- Determine **elemental composition** of organic growth media using certified reference materials for calibration
- Compare performance **with Inductively Coupled Plasma techniques**

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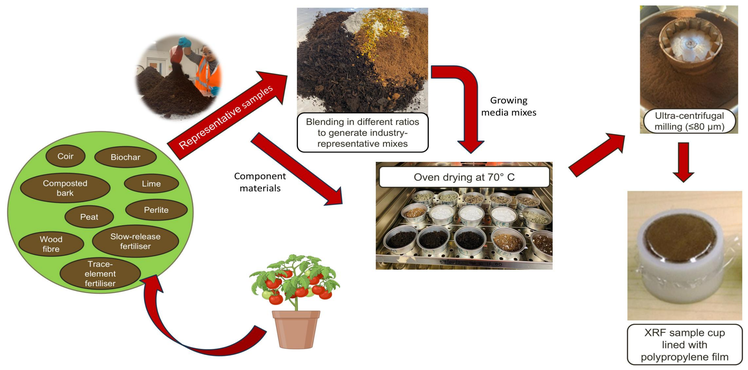
METHODOLOGY

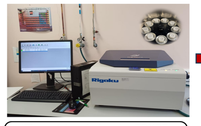
Sample Preparation (Loose powder)



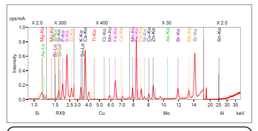
EDXRF Analysis (FP Method)

Selection of Test Samples

- QC-50 Mix (50% peat, 30% wood fibre, 10% composted bark, 10% coir + fertiliser + lime)
- 100% Peat



Elemental analysis using EDXRF spectrometer (FP method with a plant-based organic materials library)



Comparison of concentrations against ICP-OES reference measurements.

Large discrepancies compared with ICP-OES; therefore, we decided to develop an empirical calibration approach to improve accuracy.

Fundamental parameters a theory-based, standardless calibration method built into the instrument.

EDXRF Analysis (Empirical Calibration)

Selection of Calibration Standards
18 matrix-matched standards
(CRMs, in-house growing media, individual component standards)

→ Development of Initial Empirical Calibration Model

→ Iterative Optimisation of Calibration
- Validation sample: QC-50
- Model refined until relative differences <15% for all elements

→ Application of finalised calibration model to Independent Sample
- 100% peat

→ Validation of Performance
- Comparison with ICP-OES reference measurements

$$\text{Relative Difference}(\%) = \frac{(EDXRF - ICP)}{ICP} \times 100$$

Empirical calibration is a method where we build a relationship between instrument signal and known concentrations using real standards.

RESULTS

EDXRF Analysis (FP Method)

QC 50	Concentration (mg/kg) - EDXRF	Concentration (mg/kg) - ICP	Relative Difference (%)
Phosphorus	901333.33	2432.05	36961
Potassium	40666.67	8219.55	395
Magnesium	ND	2537.08	ND in EDXRF
Calcium	21200.00	23905.14	-11
Sulfur	16266.67	4424.76	268
Iron	6030.00	810.67	644
Manganese	1200.00	153.66	681
Zinc	276.67	42.18	556
Copper	236.67	30.15	685
Molybdenum	119.50	8.86	1249
Chlorine	3640.00	ND	ND in ICP

Peat	Concentration (mg/kg) - EDXRF	Concentration (mg/kg) - ICP	Relative Difference (%)
Phosphorus	658500.00	168.77	390084
Potassium	ND	58.23	ND in EDXRF
Magnesium	18150.00	2037.70	791
Calcium	32000.00	3557.20	800
Sulfur	97550.00	2879.60	3288
Iron	69200.00	712.31	9615
Manganese	3115.00	27.00	11437
Zinc	1002.50	5.90	16901
Copper	669.00	2.52	26483
Molybdenum	344.00	1.86	18411
Chlorine	46950.00	ND	ND in ICP

A relative difference of ≤15% was used as the threshold for satisfactory agreement between ICP and EDXRF measurements

- These results confirm that the FP approach combined with loose-powder preparation is unsuitable for accurate elemental analysis of organic growing media.

EDXRF Analysis (Empirical Calibration)

Iterative Optimisation of Calibration
- Validation sample: QC-50
- Model refined until relative differences <15% for all elements

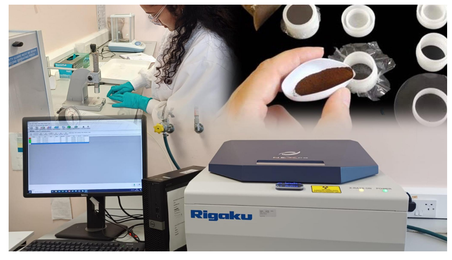
QC 50	Concentration (mg/kg) - EDXRF	Concentration (mg/kg) - ICP	Relative Difference (%)
Phosphorus	2270.00	2432.05	-7
Potassium	7975.00	8219.55	-3
Magnesium	2210.00	2537.08	-13
Calcium	22350.00	23905.14	-7
Sulfur	4815.00	4424.76	9
Iron	748.50	810.67	-8
Manganese	159.50	153.66	4
Zinc	39.70	42.18	-6
Copper	30.45	30.15	1
Molybdenum	8.45	8.86	-5
Chlorine	733.50	ND	ND in ICP

Peat	Concentration (mg/kg) - EDXRF	Concentration (mg/kg) - ICP	Relative Difference (%)
Phosphorus	216.50	168.77	28
Potassium	40.65	58.23	-30
Magnesium	2245.00	2037.70	10
Calcium	3575.00	3557.20	1
Sulfur	3415.00	2879.60	19
Iron	961.50	712.31	35
Manganese	36.60	27.00	36
Zinc	7.75	5.90	31
Copper	4.60	2.52	83
Molybdenum	3.64	1.86	96
Chlorine	708.00	ND	ND in ICP

- When applied to 100% peat, agreement with ICP-OES results improved substantially for some macro-element but remained poor for most trace elements.
- **Matrix matching** is challenging because mixes are mostly organic by volume, yet variable inorganic amendments (e.g., lime, fertilisers, and sometimes perlite) can strongly influence XRF absorption.

CONCLUSIONS

- **Matrix-matched calibration improved macro-element accuracy**, but trace-element performance and transferability remain limited.
- **Pressed-pellet preparation** is expected to **enhance sample homogeneity** and **calibration consistency** compared to loose powder.
- **ICP-OES validation using ashed samples** will increase **accuracy** and **reduce matrix effects**.



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