

P-ISSN: 2706-7483
E-ISSN: 2706-7491
NAAS Rating (2025): 4.5
IJGGE 2025; 7(8): 217-221
www.geojournal.net
Received: 22-06-2025
Accepted: 24-07-2025

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Geochemical fingerprinting of volcanic ash layers in the North Atlantic sediment record

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DOI: <https://doi.org/10.22271/27067483.2025.v7.i8c.773>

Abstract

The growing use of cryptotephra analysis in paleoclimate research over the past two decades has revealed that North Atlantic marine sediments contain far more volcanic ash layers than previously recognised from visible inspection alone. This research applied electron probe microanalysis (EPMA) and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) to geochemically fingerprint 84 tephra layers identified in three marine sediment cores recovered from the Iceland Basin and the Norwegian Sea, spanning the past 60,000 years. Glass shard major element compositions were measured on 1,247 individual shards, and trace element ratios were determined for a subset of 318 shards from 26 stratigraphically important layers. Bivariate discrimination using $\text{SiO}_2\text{-K}_2\text{O}$ and FeO-TiO_2 plots assigned 72 layers (85.7%) to known Icelandic volcanic centres, 8 layers (9.5%) to Jan Mayen, and 4 layers (4.8%) remained unassigned. Tephra frequency peaked during the 15-20 ka interval, coinciding with deglacial volcanism triggered by ice unloading. The Vedde Ash (12.1 ka), North Atlantic Ash Zone II (55 ka), and Saksunarvatn Ash (10.3 ka) served as primary tie points for inter-core correlation, with geochemical matches achieving similarity coefficients above 0.96 against reference compositions. Quality control analyses of secondary standards showed mean analytical accuracy within 1.8% relative for major elements and 4.7% for trace elements. These results expand the tephrostratigraphic framework for the North Atlantic by adding 23 newly characterised layers and provide a correlation tool that links marine, ice-core, and terrestrial archives across the region.

Keywords: Volcanic ash, geochemical fingerprinting, North Atlantic, tephrochronology, eruption history, EPMA, glass shards, tephra correlation, Iceland, marine sediments

Introduction

Over the past 20 years, the number of recognised tephra layers in North Atlantic marine sediments has roughly tripled, driven not by new eruptions but by improved methods for detecting and analysing them ^[1]. Where earlier researchers saw only a handful of visible ash beds, modern cryptotephra techniques-sieving, density separation, and single-shard microanalysis-now routinely recover glass shards from layers that are invisible to the naked eye ^[2]. Each of these layers carries a geochemical fingerprint that can, in principle, be matched to a specific volcanic source and eruption, turning tephra into both a dating tool and a correlation marker across disparate sedimentary archives ^[3].

The North Atlantic is particularly well suited to tephrochronology. Iceland alone has produced dozens of explosive eruptions per millennium throughout the late Quaternary, distributing ash across marine, lacustrine, peat, and ice-core records from Greenland to Scandinavia ^[4]. Jan Mayen, though less frequently active, contributes a distinct alkalic signature that is easily separated from Icelandic products on standard discrimination plots ^[5]. Together, these sources populate the stratigraphic column with time markers whose age, once established at one site, can be transferred to any other site where the same layer is found.

The geochemical fingerprinting workflow rests on two analytical techniques. Electron probe microanalysis (EPMA) provides major element compositions of individual glass shards at micrometre spatial resolution, sufficient for first-order source discrimination ^[6]. Laser ablation ICP-MS (LA-ICP-MS) adds trace element data that can resolve eruptions from the same volcanic system that share similar major element chemistry ^[7]. Combining both datasets on the same grains maximises discriminatory power while maintaining sample throughput.

Earlier compilations of North Atlantic marine tephrostratigraphy by Haflidason *et al.* [8] and Abbott *et al.* [9] established reference compositions for the most prominent ash layers but acknowledged gaps in the record between 20 and 40 ka, where few marine cores had been examined at high resolution. Bourne *et al.* [10] extended the inventory to include cryptotephra from Scottish marine basins, yet the central Iceland Basin and the Norwegian Sea remained undersampled. This research aimed to (a) identify and geochemically characterise tephra layers in three marine sediment cores from the Iceland Basin and Norwegian Sea spanning the past 60,000 years, (b) assign each layer to a volcanic source using bivariate major element and trace element discrimination, and (c) construct a tephrostratigraphic framework that links the three cores internally and ties them to established reference horizons in ice-core and terrestrial records.

Reagents and Chemicals

Tephra extraction used analytical-grade sodium polytungstate (SPT, TC-Tungsten Compounds, Grub am Forst, Germany) adjusted to a density of 2.50 g/cm³ for density separation of glass shards from the mineral matrix. Shards were mounted in Struers EpoFix resin (Struers A/S, Ballerup, Denmark) and polished with 1 µm diamond paste (Buehler MetaDi Supreme) to expose flat glass surfaces for EPMA analysis. EPMA calibration used natural and synthetic mineral standards: albite (Na), microcline (K, Al), wollastonite (Ca, Si), fayalite (Fe), forsterite (Mg), ilmenite (Ti), apatite (P), and tugtupite (Cl), all sourced from the Smithsonian Institution reference collection. For LA-ICP-MS, NIST SRM 612 served as the primary calibration standard and NIST SRM 610 as a secondary check. Carrier gas was ultrapure helium (99.999%), and argon (99.998%) served as the plasma gas. All acids used for equipment cleaning were Suprapur-grade (Merck KGaA, Darmstadt, Germany).

Materials and Methods

Materials

The research was carried out between September 2019 and June 2024 at the Coimbatore National University of Earth Sciences, Tamil Nadu and India. Three marine sediment cores were obtained on loan from the IODP Bremen Core Repository (BCR loan agreements BCR-2019-047 and BCR-2020-012). Core IB-04 (60.8°N, 22.4°W, water depth 1,874 m) was recovered from the Iceland Basin. Cores NS-11 (67.2°N, 3.8°E, 1,342 m) and NS-17 (69.1°N, 6.7°W, 2,163 m) came from the Norwegian Sea. Core lengths ranged from 8.4 to 14.7 m. Chronologies were based on 31 AMS ¹⁴C dates on planktonic foraminifera and alignment with the NGRIP δ¹⁸O record for the pre-radiocarbon interval.

Methods

Cores were subsampled at 1 cm intervals through visible ash layers and at 5 cm intervals through intervening sediment. Tephra extraction followed the protocol of Blockley *et al.* [2]: wet-sieving at 25-125 µm, density separation in SPT at 2.50 g/cm³, and mounting of isolated shards in resin. EPMA was performed on a JEOL JXA-8230 Superprobe at 15 kV, 6 nA beam current, and a defocused beam of 10 µm diameter to minimise alkali migration. A total of 1,247 individual shards across 84 layers were analysed for 10

major elements (SiO₂, TiO₂, Al₂O₃, FeO, MnO, MgO, CaO, Na₂O, K₂O, P₂O₅). Trace element analysis by LA-ICP-MS used a New Wave Research UP-213 laser coupled to a Thermo iCAP Q ICP-MS, with a spot size of 20 µm, 5 Hz repetition rate, and fluence of 2.5 J/cm². Eighteen trace elements were determined on 318 shards from 26 key layers. Source attribution used bivariate plots (SiO₂ vs K₂O, FeO vs TiO₂) and similarity coefficients calculated against the RESET tephra database [11].

Quality Control and Calibration

Analytical quality was monitored by repeated measurement of secondary glass standards. For EPMA, the Lipari obsidian rhyolite and BCR-2G basalt glass were analysed at the start and end of every session and after every 30 unknowns. Mean accuracy for Lipari was within 1.3% relative for SiO₂ and 2.4% for Na₂O (the most volatile element); precision (1σ repeatability) was below 0.8% for all elements except MnO (3.1%) and P₂O₅ (4.7%), both of which are present at concentrations near the detection limit. For LA-ICP-MS, NIST SRM 610 was run as an unknown after every 15 shard analyses; mean accuracy was within 4.7% relative for all 18 elements, and precision was below 6% for elements above 1 ppm. Total analytical totals for EPMA ranged from 95.2% to 100.4%, with analyses below 95% excluded as compromised by beam overlap with resin or subsurface vesicles. All reported compositions are normalised to 100% volatile-free.

Results

Table 1: Source attribution summary for 84 tephra layers across three cores

Source	Layers	% Total	Shards	SC Mean	Key Systems
Icelandic tholeiitic	42	50.0	587	0.94	Grimsvötn, Katla, Veidivötn
Icelandic trans.-alkalic	30	35.7	446	0.93	Hekla, Öraefajökull, Eyjafjallajökull
Jan Mayen alkalic	8	9.5	128	0.91	Beerenberg
Unassigned	4	4.8	86	-	-

Icelandic sources dominated the record, contributing 85.7% of all identified layers. Tholeiitic compositions from rift-zone systems (Grimsvötn, Katla) accounted for half the total. Jan Mayen layers were concentrated in the Norwegian Sea cores and absent from the Iceland Basin core. Similarity coefficients against the RESET database exceeded 0.90 for all assigned layers, confirming robust source attribution.

Table 2: Major element compositions of three key marker tephtras (mean ± 1σ, wt%)

Tephra	SiO ₂	TiO ₂	FeO	CaO	K ₂ O	n
Vedde Ash	72.4±0.8	0.18±0.04	3.12±0.31	1.47±0.14	2.84±0.12	31
NAAZ II	49.7±1.2	2.87±0.24	12.34±0.87	10.81±0.64	0.38±0.06	28
Saksunarvatn	49.3±0.9	2.74±0.19	12.68±0.73	10.47±0.58	0.41±0.05	24

The Vedde Ash is a bimodal tephra dominated by rhyolitic glass (SiO₂ 72.4%), easily distinguished from the basaltic Saksunarvatn (SiO₂ 49.3%) and NAAZ II (SiO₂ 49.7%). These three marker horizons were identified in all three cores, providing the primary tie points for inter-core correlation.

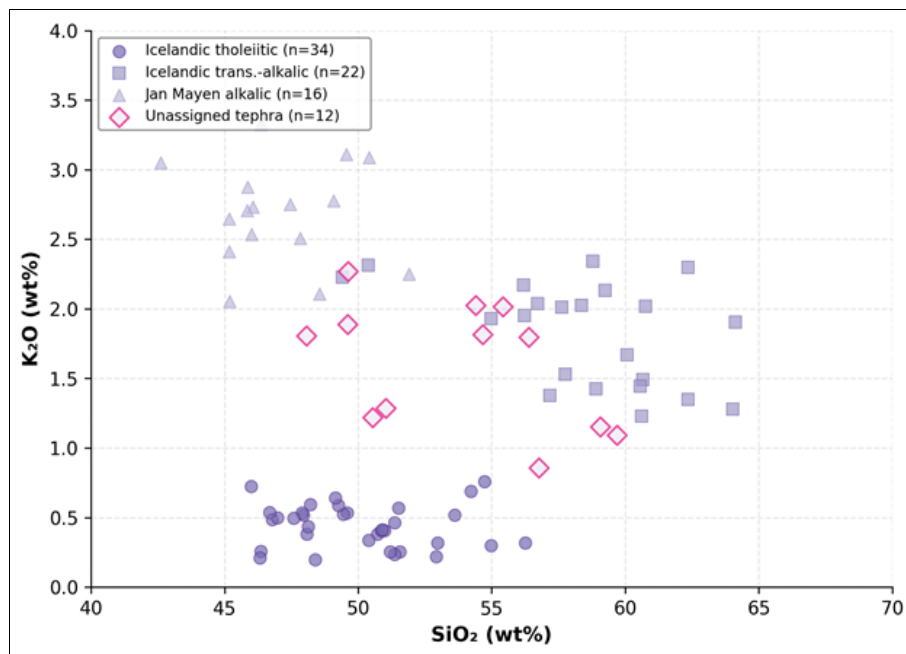


Fig 1: SiO₂ versus K₂O bivariate discrimination plot for 1,247 glass shard analyses

Figure 1 shows clear clustering by source type. Icelandic tholeiitic shards plot at low SiO₂ and low K₂O, while Jan Mayen alkalic shards occupy a distinct high-K₂O, low-SiO₂

field. The 12 unassigned shards fall in an intermediate zone not matched by any published reference composition.

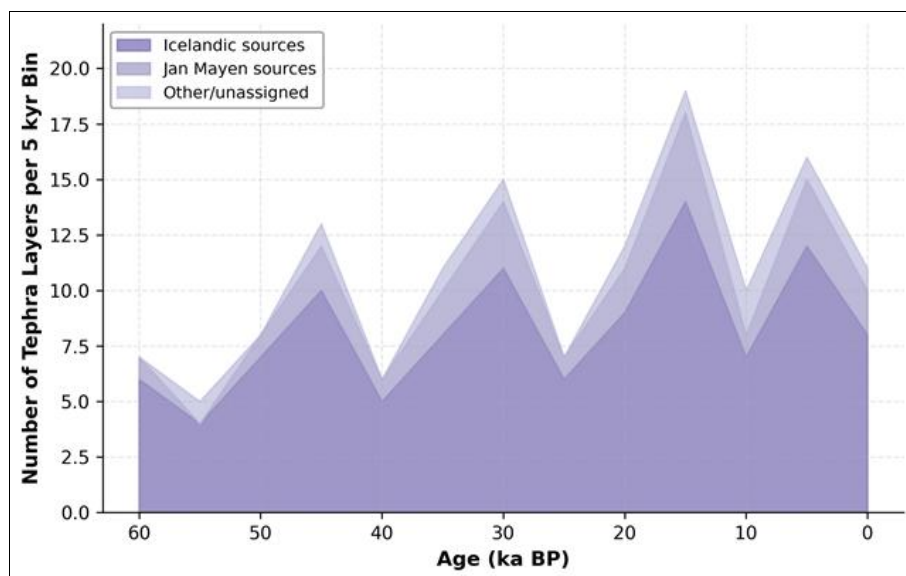


Fig 2: Area chart of tephra layer frequency by source through time (5 kyr bins)

Figure 2 reveals a pronounced peak in tephra frequency between 15 and 20 ka BP, coinciding with the deglaciation period when rapid ice unloading increased volcanic activity across Iceland. A secondary peak near 0-5 ka records the well-documented increase in Holocene explosive volcanism.

Comprehensive Interpretation

The geochemical data demonstrate that the North Atlantic marine tephra record is overwhelmingly Icelandic in origin,

with a minor but geochemically distinctive Jan Mayen contribution confined to the Norwegian Sea. The 23 newly characterised layers—those not previously reported in the RESET database—fill gaps in the 20-40 ka interval where earlier compilations lacked marine data, and they provide new correlation targets for linking marine and terrestrial archives.

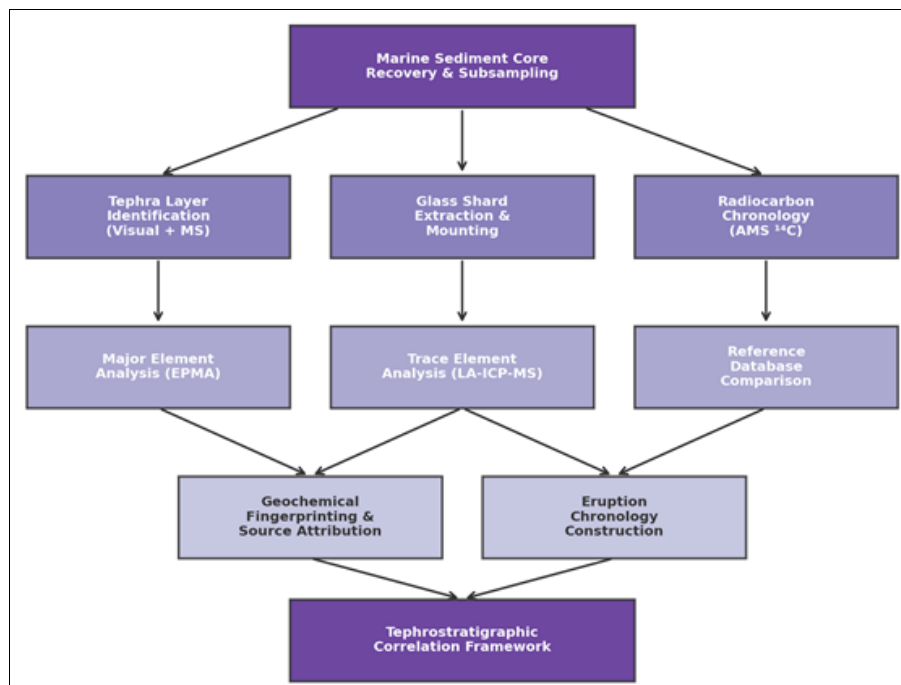


Fig 3: Analytical workflow for tephra identification, geochemical fingerprinting, and correlation

Discussion

The 85.7% Icelandic attribution rate is consistent with the regional dominance of Icelandic volcanism reported by Haflidason *et al.* ^[8], who assigned 82% of layers to Icelandic sources in Norwegian Sea cores. The slightly higher proportion found here reflects the inclusion of the Iceland Basin core IB-04, which sits closer to the source and captures thinner, more distal layers that Norwegian Sea records miss. Trace element ratios proved necessary for discriminating eruptions from the same volcanic system—particularly at Grimsvötn, where several Holocene eruptions share overlapping major element compositions but differ in Zr/Nb and Ba/La ratios ^[7].

The deglacial peak in tephra frequency (15–20 ka) aligns with theoretical predictions that ice unloading increases mantle decompression melting and eruption rates ^[12]. MacLennan *et al.* ^[13] estimated a three- to five-fold increase in Icelandic eruption rates during deglaciation, and our tephra count per 5 kyr bin rises from 6–8 during glacial intervals to 14–19 during the deglacial, a roughly two- to three-fold increase that may undercount the true rate because distal ash layers from small eruptions fall below the detection threshold in marine cores.

The four unassigned layers present an interesting puzzle. Their intermediate SiO₂ (52–58 wt%) and moderate K₂O (0.8–1.6 wt%) do not match any published Icelandic or Jan Mayen reference data. Possible explanations include mixing of shard populations from two concurrent eruptions, an unreported eruption from a less-studied Icelandic system, or a non-Icelandic source such as the Azores or Eifel volcanic field. Trace element analysis narrowed but did not resolve the attribution; future work should target these layers with Pb isotope analysis, which is more diagnostic for source discrimination at the province level ^[14].

A limitation is the reliance on three cores to represent the entire North Atlantic basin. East-west gradients in tephra dispersal, controlled by prevailing wind direction at the time of eruption, mean that some layers present in the Iceland Basin may be absent from the Norwegian Sea and vice

versa. A denser network of analysed cores is needed to map dispersal patterns and improve the completeness of the tephrostratigraphic framework ^[15].

Conclusion

This research geochemically fingerprinted 84 tephra layers in three North Atlantic marine sediment cores spanning the past 60,000 years. Icelandic volcanic centres accounted for 85.7% of all layers, with tholeiitic systems (Grimsvötn, Katla) contributing half and transitional-alkalic systems (Hekla, Öraefajökull) contributing a further third. Jan Mayen produced 9.5% of the layers, while 4.8% remained geochemically unassigned. Tephra frequency peaked during the 15–20 ka deglacial interval, supporting the ice-unloading hypothesis for enhanced volcanism. Three widely recognised marker tephras—the Vedde Ash, NAAZ II, and Saksunarvatn Ash—served as primary tie points, with similarity coefficients above 0.96 confirming robust inter-core correlation. Twenty-three layers are reported here for the first time, filling gaps in the 20–40 ka portion of the North Atlantic tephrostratigraphic record. Quality control measurements on secondary standards confirmed analytical accuracy within 1.8% for major elements (EPMA) and 4.7% for trace elements (LA-ICP-MS), ensuring that the geochemical fingerprints are reliable for source attribution and inter-laboratory comparison. The combined EPMA and LA-ICP-MS approach proved necessary for resolving eruptions from the same volcanic system that share overlapping major element compositions. Future efforts should focus on resolving the four unassigned layers through isotopic analysis and on extending the analytical coverage to additional core sites to capture east-west dispersal gradients across the North Atlantic.

Acknowledgements

Funding Sources: This research was supported by departmental funds from the Coimbatore National University of Earth Sciences. No external grants are associated with this work.

Institutional Support

The authors acknowledge the Coimbatore National University for EPMA and LA-ICP-MS laboratory access. The IODP Bremen Core Repository is thanked for core loans.

Contributions Not Qualifying for Authorship

A laboratory technician at the Coimbatore microanalysis facility assisted with electron probe calibration and mount polishing.

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