



SEM-based methods for the analysis of basaltic ash from weak explosive activity at Etna in 2006 and the 2007 eruptive crisis at Stromboli

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ABSTRACT

We present results from a semi-automated field-emission scanning electron microscope investigation of basaltic ash from a variety of eruptive processes that occurred at Mount Etna volcano in 2006 and at Stromboli volcano in 2007. From a methodological perspective, the proposed techniques provide relatively fast (about 4 h per sample) information on the size distribution, morphology, and surface chemistry of several hundred ash particles. Particle morphology is characterized by compactness and elongation parameters, and surface chemistry data are shown using ternary plots of the relative abundance of several key elements. The obtained size distributions match well those obtained by an independent technique. The surface chemistry data efficiently characterize the chemical composition, type and abundance of crystals, and dominant alteration phases in the ash samples. From a volcanological perspective, the analyzed samples cover a wide spectrum of relatively minor ash-forming eruptive activity, including weak Hawaiian fountaining at Etna, and lava-sea water interaction, weak Strombolian explosions, vent clearing activity, and a paroxysm during the 2007 eruptive crisis at Stromboli. This study outlines subtle chemical and morphological differences in the ash deposited at different locations during the Etna event, and variable alteration patterns in the surface chemistry of the Stromboli samples specific to each eruptive activity. Overall, we show this method to be effective in quantifying the main features of volcanic ash particles from the relatively weak – and yet frequent – explosive activity occurring at basaltic volcanoes.

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1. Introduction

The emission of volcanic ash is a common occurrence and can have far reaching effects – as clearly exemplified in April 2010 with the travel crisis in Europe caused by activity at Eyjafjallajökull volcano in Iceland. Globally, eruptions including those of much lower intensity than at Eyjafjallajökull eject > 1,000,000 m³ of ash into our atmosphere on a monthly basis (Simkin and Siebert, 2000), often affecting water supplies, crops, air and road traffic and climate on a local to international scale. Though a nuisance to humans, its small particle size, low terminal velocity and widespread distribution makes ash the safest and simplest volcanic particle to collect in real-time. Within the last decade, studies have linked variations in the componentry of ash emitted during explosive eruptions to

changing eruptive styles (Andronico et al., 2005, 2009a, 2009b; Taddeucci et al., 2002), and used the ejection of juvenile ash as a precursor to larger explosive eruptions (Cashman and Hoblitt, 2004; Watanabe et al., 1999).

Yet still, there is a paucity of published studies focused on characterizing the textural properties of ash (relative to lapilli and bombs), probably because its small size makes ash inherently difficult to analyze. The studies that do exist highlight a long-standing gap between grain-size/componentry analysis (e.g. Cas and Wright, 1987; De Rosa, 1999; Fisher and Schmincke, 1984), and scanning electron microscope (SEM) studies (e.g. the pioneering works of Heiken and Wohletz, 1985; Sheridan and Marshall, 1983; Wohletz, 1987). The former analyses are generally conducted using sieves, automated particle analyzers, and/or binocular microscopes. These studies provide fast, statistically robust information on a large number of particles, but lack detailed shape and morphological information. SEM studies in general provide detailed information, but on only a small number of particles within a sample.

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Recent progress has been made toward automating methods to obtain grain size and particle morphology data for ash samples (Dellino and La Volpe, 1996; Dellino and Liotino, 2002; Ersoy, 2010; Ersoy et al., 2007; Kueppers et al., 2006; Maria and Carey, 2002; Riley et al., 2003). However the attainment of automated, quantitative data for the surface chemistry of volcanic particles until recently remained limited to volcanic aerosols (Martin et al., 2008), despite the wide application and usefulness of surface chemistry data, e.g. in aiding the establishment of component classes (Cioni et al., 2008), in tephrostratigraphy (Sulpizio et al., 2008), in unraveling eruption plume chemistry (Delmelle et al., 2007), which has further implications for the health and environmental aspects of ash transport and deposition (Horwell et al., 2003).

Italy's Istituto Nazionale di Geofisica e Vulcanologia (INGV) is in charge of monitoring active and quiescent volcanoes nationally. Recently, scientists developed ash collection and analysis tools specifically suitable for eruption monitoring, mainly at Etna and Stromboli volcanoes (Andronico et al., 2009a; Taddeucci et al., 2002). Such techniques enable largely automated, relatively quick, and quantitative classification of the morphoscopy and surface chemistry of a large number (hundreds) of ash particles using a SEM equipped with particle analysis software (Taddeucci et al., 2007).

Here we test the effectiveness and robustness of such methods on multiple ash samples from two study cases: an explosive, Strombolian to Hawaiian, episode at Etna on 24 November 2006 (Andronico et al., 2009b), and the February–April 2007 eruptive crisis of Stromboli (Barberi et al., 2009; Mori and Burton, 2009; Calvari et al., 2010). The latter includes ash from lava–sea water interaction, the 15 March paroxysm, and weaker Strombolian explosions at the summit craters. Our data show that the methods are efficient in characterizing and distinguishing ash particles collected at different locations (Etna case), and from different sources (Stromboli case), and also give insight into the particle source and eruptive dynamics at both volcanoes. In particular, the different sources of ash at Stromboli have distinctive alteration signatures, while the Etna samples highlight subtle differences in grain size and componentry. This and future studies focused on ash at basaltic volcanoes are seminal given that the fragmentation processes leading to ash from Hawaiian and Strombolian style explosions are unclear (Patrick et al., 2007).

2. Methods

Sampling strategies employed at each volcano are described below. All ash particles were analyzed in the state they were collected, i.e. without washing or chemical treatment, and without any sorting by grain size. The Etna samples consisted of only several hundred grains, so the complete sample was analyzed. The Stromboli samples each consisted of thousands of ash particles, from which a subset of 500–1000 was randomly selected by scooping a fraction of ash from the container that housed the entire 'randomly-sorted' sample. Ash grains were mounted on a 3 mm piece of double sided carbon tape adhered to a metal stub, then loaded into the SEM. The JEOL JSM 6500F Field Emission (Schottky-type) scanning emission microscope (FE-SEM) at the INGV-Roma was used to obtain data regarding texture (vesicularity), morphoscopy, and surface chemistry of ash grains, with the latter two parameters obtained using the Particle Analyzer tool of the JED 2200 software package. In general, the complete analysis of one sample took 3–5 h on the SEM.

2.1. High resolution imaging

In comparison to conventional SEMs, FE-SEMs offer a more stable electron source and a smaller beam capable of higher spatial

resolution at a lower acceleration voltage. The nominal resolution of the FE-SEM at INGV-Roma is 1.5 and 3 nm at 15 and 1 kV voltage acceleration, respectively. Magnifications up to 100,000 \times were used to visualize sub-micron size features of the ash. The carbon coating was visible at higher magnifications such that use of a noble metal coating was necessary.

2.2. Morphoscopy

Morphoscopic data were obtained by first setting the brightness and contrast of the FE-SEM so that ash particles appeared bright against the dark background of the sample holder. The Particle Analyzer software was then programmed to automatically acquire backscattered electron (BSE) images over the entire sample holder. As a compromise between image resolution and acquisition time, images were acquired at 512 \times 384 pixels, and at a single magnification chosen such that it was low enough to include the largest particles in a single image but high enough to measure the smallest particles. Within each image, the area, perimeter, rectangularity, elongation, circularity, compactness, Feret (maximum) diameter and Heywood (equivalent) diameter of each particle was automatically calculated – generally for a total of 500–1000 particles. In this study we report results for the parameters of compactness, elongation, and Heywood diameter. Compactness was calculated as the ratio between the area of the minimum rectangle circumscribed by the particle and the particle area, with a non-dimensional value between 0 and 1 (1 being a square). Irregular and rounded particle edges decrease the compactness parameter. Elongation was calculated as the square of the longest particle segment divided by the particle area (Dellino and La Volpe, 1996), such that the more elongate the particle, the higher the elongation parameter. Heywood diameter was calculated from the particle area as the diameter of a circle with the same area. The morphoscopic analyses were determined to be orientation invariant through repeat analysis on rotated particles. As a test for the effectiveness of FE-SEM based grain size distribution analysis we also performed, on duplicate samples, grain size analyses by an automated, digital image processing-based particle size analyzer (CAMSIZER[®], see <http://www.retsch-technology.com/rt/products/digital-image-processing/camsizer/function-features/> for more information) at INGV-Sezione di Catania.

2.3. Surface chemistry

Surface chemistry analyses were conducted using the energy dispersion system (EDS) of the FE-SEM. Within the Particle Analyzer software and using images obtained in the morphoscopic analyses, a rectangular area was defined on >100 randomly chosen particles. An X-ray spectrum from the surface of each particle within the rectangle was then automatically acquired and converted into a standardless quantitative chemical analysis for specified elements. Since the X-ray spectrum was obtained over a large portion of the particle surface, the corresponding analysis is an average of the phases cropping out in the scanned area, possibly including pristine glass, crystals, and alteration phases in variable proportions. This technique provides "bulk" information on the glass composition, crystallinity and degree of secondary alteration of the particle (Taddeucci et al., 2007) and was used to effectively characterize the chemical composition of magnetic particulate matter in the city of Rome (Sagnotti et al., 2009).

The analytical error associated with spectrum quantification is within 10% of the measured value (5% for oxides with abundances >10% mass tot.), as deduced by repeated analyses and a comparison with electron microprobe analyses performed on basaltic glasses (see Appendix A). A larger and less-explored source of error is the effect of particle roughness and orientation relative to the

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