

APPLICATION OF ELECTRON MICROPROBE TECHNIQUE TO THE CHARACTERIZATION OF VOLCANIC SANDS

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ABSTRACT

A large number of single-grain analyses, which demand a careful planning and proper selection of samples for optimum results, permitted the distinction between minerals of different chemical composition in the same sample thus contributing towards their characterization. A variety of minerals in the volcanic sediments of the Guadeloupe archipelago (French West Indies) were identified. These include chiefly labradorite, bytownite, hypersthene, augite, magnetite, hornblende and acid volcanic glass. Andesine, anorthite, ilmenite, olivine and quartz are of minor importance. This mineral assemblage stratified within insular margin sediments, represents a calc-alkaline mineralogy and resembles volcanic products of Basse Terre (western Guadeloupe).

Key-words: Electron microprobe technique volcanic sands, Guadeloupe archipelago, French West Indies.

INTRODUCTION

Marine volcanic sands in regions bordering island arcs, have been widely reported in recent years. Mineralogy and chemical composition of pyroclastic sands in the vicinity of active volcanoes can lead to its correlation with terrestrial sources thus providing vital information concerning the nature and history of volcanism on the island arc. The advent of the electron microprobe has further intensified studies on volcanic sands, often found intercalated within the sedimentary cover. Based on the chemical composition of volcanic glass, Federman and Carey (1980) and Keller (1981) identified and correlated deep sea ash layers with terrestrial sources and specific eruptions of particular volcanoes. Smith, Ledbetter and Ciesielski (1983) distinguished tholeiitic and calc-alkaline series. Murat (1984) and Ledbetter (1985) classified and correlated volcanic layers to known volcanic episodes. But these studies mainly describe volcanic glass with little information on other mineral components. Sibley and Pentony (1978) reported feldspar and quartz, showed multiple source rocks and indicated an increasing volcanism on the adjacent island arc. Monaco, Valette, Hoffert and Picot (1979) characterized volcanic glass, pyroxenes and plagioclases and attributed their occurrence to various factors operating in the region. Based on a detailed account of feldspars, Travena and Nash (1981) identified and classified different groups of feldspars from eight different sources. Maynard (1984) published microprobe results of plagioclase in deep sea sands and

distinguished different geological settings on a worldwide scale. The present paper presents some aspects on the characterization of marine volcanic sands using electron microprobe technique. Representative analyses of each mineral type are described with some practical details; a probable source is also indicated.

Geology of the area

Mineralogy, geochemistry and volcanism of Guadeloupe island is well documented (Westercamp and Mervoyer, 1976; Brown, Holland, Sigurdsson, Tomblin and Arculus, 1977; Blanc, 1983). The island, bounded by the Atlantic ocean on the east and the Caribbean sea on the west, is subdivided into two parts: Grande Terre, the eastern portion, is a low-lying flat zone entirely covered by calcareous deposits. Basse Terre, the western part, principally consists of volcanic formations (and related soils) with the active volcano "La Soufriere" in the southern extremity. Several explosive eruptions occurred in the past and volcanic activity persists at present. Andesites and dacites of calc-alkaline nature are the main rock types exposed on Basse Terre (Fig. 1).

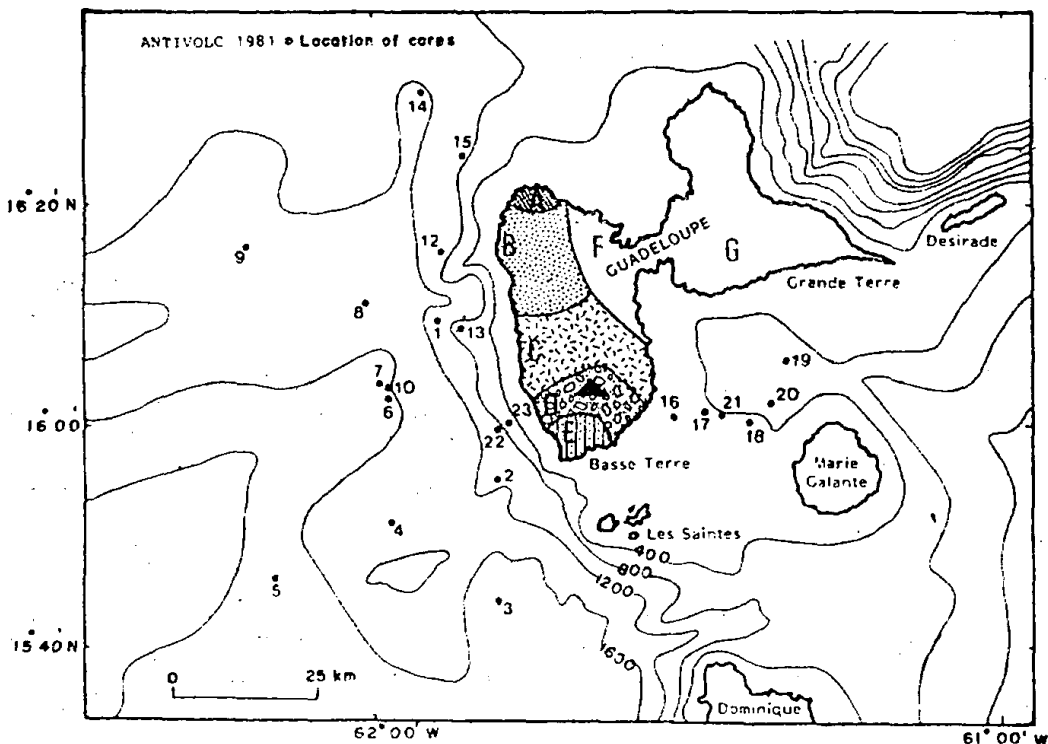


Fig. 1. Location of cores collected in the Guadeloupe archipelago. The geology of the island is represented as A-Hornblende andesites; B-Calc-alkaline rocks, dacites and andesites; C-Acid andesite flows; D-Recent calc-alkaline rocks with hypersthene and augite. Solid triangle represents "La Soufriere" active volcano; E-Olivine basalts; F-Altered zone; G-Calcareous deposits of marine origin (Data from Westercamp and Mervoyer, 1976).

MATERIALS AND METHODS

As a part of the French national project P.I.R.P.S.E.V., cores were collected from the Guadeloupe archipelago (French West Indies) in water depths ranging from 200-1800m during the oceanographic expedition "Antivolo 81" on board "N/O *Entrecasteaux*" of the French Navy (Fig. 1). This was a joint project between Institut de Physique du Globe (France), L.S.G.M., University of Perpignan (France) and Istituto Universitario Navale (Italy). Several cores contain sand layers.

Sample selection and preparation of sections

Cores were sampled every 2 or 3 cm depending upon colour, texture and presence of sand layers. Each sample was wet-sieved through a 40 micron sieve and only the fraction $> 40 \mu m$ was utilised. Coarse pyroclastic sands were examined under a binocular microscope to identify various components present; those showing a variety of minerals were finally selected and further separated to obtain a fraction between 150 and 2000 μm . The upper limit of 2 mm excludes undesired rock fragments. Selected samples were then attacked with 1:1 HCl to eliminate carbonate debris which was often found present. Pure mineral sands were washed several times in distilled water and dried. A significant portion was embedded in a resin (epoxy), according to a method perfected by the L.S.G.M. (University of Perpignan) and adopted by Aloisi and Monaco (1980). Air bubbles, which cause analytical problems, were removed by vacuum suction. Polishing of impregnated minerals was accomplished in four or five steps on an electrical polishing machine using 2.0.5 and 0.1 micron alumina powder slurry. Between each polishing step, the samples were ultrasonically cleaned and dried. Care was taken to obtain a flat and a perfectly smooth mineral surface, a basic requirement for microprobe work. Further details are discussed by Keil (1973).

Elemental analyses were carried out on a CAMEBAX electron microprobe at the "Laboratoire de Microsonde Electronique, Universite des Sciences et Techniques du Languedoc, Montpellier, France. It was operated at a voltage of 15 kV, counting time was about 15 seconds and the electron beam was of the order of 1 to 3 micron diameter; its drift was checked and corrected from time to time. All standards used were synthetic compounds. Analyses include elements as Na, Mg, Al, Si, K, Ca, Ti, Fe, Mn and Cr, sufficient to ascertain the petrography and characterize each mineral. Various theoretical and practical aspects of the microprobe are explained by Andersen (1973) and Maurice, Meny and Tixier (1978). The analytical technique involved analysis of individual minerals exhibiting different physical characteristics. In general, one point analysis of 25 to 30 single grains of each polished section was obtained.

Though the microprobe technique is convenient and rapid, certain practical and analytical difficulties were encountered and they are: (1) during

vacuum suction of air bubbles from the viscous resin, a preferred orientation and differential settling of grains was observed; therefore the mixture was restirred and procedure often repeated; (2) Minerals show different physical properties, hence the polishing effect on them varies. Plagioclase has a better surface quality than magnetite which is more difficult to analyse and; (3) Pumice posed most problems. Being fragile, it exhibits poorly polished and crackled surfaces; therefore pumice could not be analysed.

RESULTS AND DISCUSSION

The microprobe, a costly technique, is an useful equipment for the analyses of marine volcanic sands. It warrants a careful planning and proper selection of representative samples for optimum results. The method permitted quick analyses and rapid distinction of minerals of different chemical composition in the same sample, thus contributing towards the characterization of pyroclastic sands which is extremely difficult and time consuming by other conventional procedures. Using this technique, a large number of analyses of 10 elements on a variety of individual grains were obtained. The most common are plagioclases, pyroxenes and magnetites. For confirmation, all analyses were compared with typical mineral compositions given by Deer, Howie and Zussman (1966). Each type is briefly described and represented in Table I.

Plagioclase

Plagioclase minerals are colourless and subhedral, often showing inclusions. This group consists of 44-58% SiO_2 ; 26-34% Al_2O_3 ; 8-18% CaO and 0.5-6% Na_2O . Based on oxide contents, 4 types of minerals can be characterized: Andesine, labradorite, bytownite and anorthite (Table I).

Labradorite and bytownite were encountered in all cores studied; the former is abundant with 12-14% CaO and 3-4% Na_2O . On an average, labradorite is composed of Or 1; Ab 35; An 64, as compared to bytownite which is more calcic, corresponding to Or 1; Ab 18; An 81. Andesine with high Na_2O (> 5%), consists of Or 3; Ab 59; An 38. Few analyses representing anorthite, showing very high CaO (up to 18%), could also be characterized; they fall in the pure anorthite field.

Percentage of Or, Ab and An for plagioclase given in Table I was obtained by recalculating normative values to 100, as suggested by Travena and Nash (1981). Position range of minerals is shown in Fig. 2. A It was observed that plagioclase is chiefly Ca bearing, with labradorite and bytownite being the most common components; andesine and anorthite are less frequent. Potassium variety, which is an indicator of volcanic provenance according to Travena and Nash (1981) and later contested by Maynard (1984), is absent in sediments studied.

TABLE 1: Composition of selected minerals, each representing a single grain, in volcanic sediments of the Guadeloupe archipelago. Figures in brackets denote sampling position in centimeters from top of each core. The order of elements as measured by the microprobe is maintained.

Core	MINERAL	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	TiO ₂	FeO	MnO	Cr ₂ O ₃	TOTAL	%
03 (52)	Andesine	6.01	0.00	26.13	57.43	0.51	8.55	0.00	0.58	0.02	0.00	99.24	Or = 3.2 Ab = 58.6 An = 38.6
		4.02	0.00	30.55	53.57	0.06	12.74	0.02	0.58	0.08	0.04	101.65	Or = 0.4 Ab = 35.1 An = 64.5
		2.09	0.00	32.88	48.38	0.04	15.66	0.00	0.67	0.00	0.00	99.72	Or = 0.3 Ab = 18.5 An = 81.2
06 (94)	Anorthite	0.59	0.03	34.90	44.47	0.01	18.66	0.00	0.55	0.00	0.22	99.42	Or = — Ab = 5.1 An = 94.8
06 (248)	Hypersthene	0.03	20.50	0.94	51.91	0.00	1.53	0.12	23.85	1.00	0.00	99.86	Ca = 3.1 Mg = 58.6 Fe = 38.3
		0.22	12.72	1.22	50.97	0.00	19.50	0.24	11.42	0.32	0.02	96.6	Ca = 42.2 Mg = 38.5 Fe = 19.3
		0.18	13.87	6.78	48.13	0.00	22.08	0.51	5.64	0.06	0.53	97.78	Ca = 48.2 Mg = 42.3 Fe = 9.5
08 (318)	Fe-Ti Oxides	0.00	2.29	2.86	0.54	0.00	0.00	11.54	76.84	0.53	0.00	94.62	—
17 (07)	Ilmenite	0.07	2.62	0.19	0.14	0.00	0.04	43.46	47.30	0.60	0.00	94.43	—
14 (231)	Hornblende	1.48	14.76	7.54	48.42	0.18	10.23	1.07	14.25	0.65	0.03	98.60	—
12 (85)	Olivine	0.02	35.07	0.00	37.75	0.01	0.23	0.02	26.58	0.58	0.23	100.49	—
19 (331)	Quartz	0.01	0.00	0.10	97.08	0.00	0.02	0.00	0.13	0.00	0.00	97.36	—

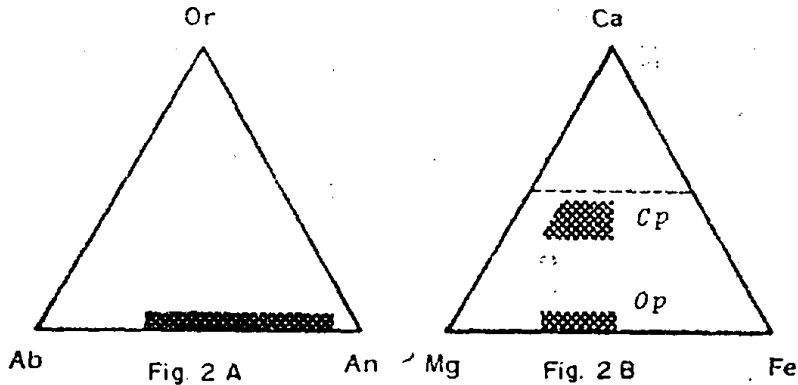


Fig. 2. Or-Ab-An (A) and Ca-Mg-Fe (B) diagrams showing the position of plagioclase and pyroxene group of minerals resp. (Op: orthopyroxenes; Cp: clinopyroxenes.)

Pyroxene

Based on the CaO, MgO and FeO contents, two varieties of pyroxenes showing compositional variations can be characterized. Those which are Mg-Fe bearing fall under the category of hypersthene whereas Ca-bearing pyroxenes are classified as augites. Hypersthene is greenish brown in colour, characterized by high MgO and FeO (up to 22% and 25% respectively). MnO is important. In comparison, augite is identified by high CaO (17-20%) and low MgO-FeO contents. In a triangular diagram, hypersthene and augite occupy two distinct positions which correspond to Ca 3, Mg 58, Fe 39 and Ca 40, Mg 40, Fe 20 (on an average) respectively. Only one diopside analysis, with low FeO content was observed (Table I).

Percentage of Ca, Mg and Fe for pyroxenes given in Table I, was calculated based on procedure given by Deer, Howie and Zussman (1966). Position range in a triangular diagram is given in figure 2 B.

Pyroxenes mainly comprise hypersthene. There is little difference between the composition of hypersthene and pigeonite; the presence of the former has been confirmed by lower CaO contents and characteristic pleochroic properties. Pigeonite has therefore been excluded. Ca pyroxenes, categorised as augites, are less abundant in sediments investigated.

Fe-Ti Oxides

Magnetite was easily identified owing to its exceptionally high FeO content of around 78%. Up to 12% TiO₂ and 2 to 3% MgO were observed. This mineral can be termed as titanomagnetite. Ilmenite composed of an equal proportion of FeO and TiO₂ (47% and 43% respectively). Only two single grains were analysed.

Magnetite was present in all samples as individual euhedral crystals and also as opaque inclusions in plagioclase and pyroxene. It has been termed as

titano-magnetite owing to its high TiO_2 content, and has not been differentiated from a titaniferous magnetite as described by Deer, Howie and Zussman (1966).

Other minerals

Hornblende exhibited a variable composition; higher contents of Al_2O_3 (6 to 10%) and TiO_2 ($> 1\%$) discriminate it from pyroxene minerals. It has been identified on the basis of literature values. *Olivine* shows a low SiO_2 content (around 37%), and high MgO and FeO ($> 35\%$ and $> 26\%$ respectively). Among the analyses obtained, its presence is negligible. *Quartz* was noticed in certain cores with a SiO_2 content invariably above 95%. The rest is attributed to impurities. Hornblende is a common primary constituent in a variety of igneous rocks but also occurs as a secondary mineral; this wide range of paragenesis results in variation in composition (Deer, Howie and Zussman, (1966). Hornblende analysed is a Ca-amphibole resembling a basaltic or a common hornblende. So far, no attempt has been made to ascertain its exact (primary or secondary) nature. Since ilmenite, olivine and quartz are very few it is presumed that these minerals are of minor importance.

Volcanic glass

Another group showing SiO_2 values dominantly above 60% was also observed. A comparison with literature values suggests that these components fall under the category of volcanic glass (Table II). This assemblage shows SiO_2 contents which range from 60 to 80%. Calculation of normative values reveals albite, anorthite, orthoclase, hypersthene and a high percentage of free quartz (13 to 53%) which indicates acid volcanic glass, a common constituent of volcanic sediments, but also suggests a rhyolitic affinity. Volcanic glass with an average of 74% SiO_2 was reported by Sigurdsson and Carey (1981) for Caribbean Sea pyroclastic sediments.

Other analyses

The microprobe's electron beam being very narrow, fine grains or very small portions of larger grains can be studied. Several point analyses on twins, mineral inclusions or contact rims were attempted; these are given purely as an illustration and represented in Table II.

Accuracy and validity of data

According to tests carried out, the precision of analyses is within $\pm 2\%$; but for magnetite, erratic totals are found. However, duplicate analyses on the same point showed excellent reproducibility. It is often observed that certain analyses show oxide totals below or above 100; for example, glass and magnetite and to a lesser extent, pyroxene and plagioclase. Low glass totals (Table II), may be due to water content confirmed by Federman and Carey (1980) and Keller (1981). Low magnetite totals (92 to 95% only) have remained doubtful; but can be due to presence of a non-determined element. This requires

further confirmation. The sum of oxides sometimes reaching 101 is attributed to instrumental drift which was corrected from time to time.

The appearance of corundum in the norms (Table II), results due to an increase of Al_2O_3 , caused by a loss of Na during probe analyses as reported by Smith, Ledbetter and Ciesielski (1983) and also discussed by Federman and Carey (1980) and Keller (1981). The validity of results, especially of multi-component sands, seems to have escaped the attention of authors, except for a brief remark by Maynard (1984). Although there is no fixed rule regarding the number of analyses to be performed, point counts of 25 to 30 different individual grains, as carried out in this study, are presumed to be statistically valid.

Table II: Composition of minerals termed as volcanic glass in pyroclastic sediments of the Guadeloupe archipelago. Figures in brackets denote sampling position in centimeters from top of each core. The order of elements as measured by the microprobe has been maintained. The lower half of the table presents the respective normative values calculated according to standard methods.

Core →	03 (52)	03 (52)	14 (231)	06 (94)	02 (34)	21 (211)	12 (85)
Na ₂ O	3.01	3.32	2.96	0.99	4.35	1.59	4.98
MgO	4.07	0.10	0.03	0.41	0.04	0.91	0.03
Al ₂ O ₃	14.33	10.78	18.58	12.49	14.18	14.59	15.11
SiO ₂	59.88	80.38	69.02	73.18	76.04	70.85	70.24
K ₂ O	1.55	1.63	1.95	1.96	0.57	1.36	5.04
CaO	5.56	1.56	4.31	2.50	3.34	3.77	0.62
TiO ₂	0.20	0.22	0.25	0.51	0.54	0.41	0.15
FeO	8.68	1.05	1.09	3.33	0.98	4.33	1.15
MnO	0.25	0.13	0.02	0.11	0.00	0.19	0.00
Cr ₂ O ₃	0.00	0.00	0.00	0.00	0.48	0.02	0.00
TOTAL	97.52	99.18	98.21	95.49	100.52	98.02	97.32
<i>Normative values</i>							
Qz	13.02	51.42	34.80	53.22	41.34	45.72	20.52
Or	8.89	9.45	11.67	11.67	3.33	7.78	30.02
Ab	25.15	27.77	25.15	8.38	36.68	13.62	41.92
An	21.12	7.78	21.40	12.51	16.68	18.62	3.05
Di	5.35	—	—	—	—	—	—
Hy	23.35	0.69	0.23	2.32	0.23	5.10	0.23
Il	0.45	0.45	0.45	0.91	1.06	0.76	0.30
He	—	1.76	1.76	4.96	0.96	6.00	2.08
Cor	—	0.80	3.67	4.08	0.30	3.60	0.30

Results of a preliminary study of insular sands are discussed by Mascarenhas (1984). Additional data permits following general remarks: 1. Volcanic sediments of the Guadeloupe archipelago are chiefly composed of Ca-rich plagioclase (with labradorite-bytownite as principal constituents) and pyroxenes

with dominant hypersthene). This assemblage, with abundant hypersthene, represents a calc-alkaline mineralogy (Keller, Ryan, Ninkovich and Altherr, 1978) and resembles andesito-dacitic volcanic rocks exposed on Basse Terre (Westercamp and Mervoyer, 1976; Brown, Holland, Sigurdsson, Tomblin and Arculus, 1977; Blanc, 1983). 2. Representation of volcanic glass on A-F-M diagram as employed by Westercamp and Mervoyer, 1976; Smith, Ledbetter and Ciesielski, 1983, shows that glasses analysed occupy two independent positions (Fig. 3), indicating a calc-alkaline and a tholeiitic assemblage, or probably, two magma types in the geological past. This phenomenon is explained

Table III: Analyses on twins and inclusions. 1 and 2: opposite "arms" of a twinned pyroxene; 3: magnetite inclusion in (2); 4: subhedral plagioclase grain; 5: magnetite inclusion in (4); 6: pyroxene; 7: a glassy rim around pyroxene (6); 8: a "siliceous aggregate" (Pumice?) with opaque minerals; inclusions could not be analysed.

	1	2	3	4	5	6	7	8
Na ₂ O	0.03	0.00	0.00	4.42	0.00	0.02	1.35	3.17
MgO	21.25	21.62	2.04	0.05	1.79	20.54	0.01	0.27
Al ₂ O ₃	0.81	0.86	2.78	29.17	2.63	0.75	5.24	12.01
SiO ₂	52.71	52.55	0.66	53.53	0.49	52.46	87.82	77.77
K ₂ O	0.00	0.00	0.00	0.12	0.00	0.00	2.02	0.54
CaO	1.53	1.35	0.03	11.73	0.05	1.40	0.18	2.80
TiO ₂	0.09	0.09	11.23	0.00	11.76	0.15	0.16	0.24
FeO	22.94	21.90	73.98	0.86	75.57	23.45	0.51	1.49
MnO	0.82	0.97	0.46	0.00	0.44	1.00	0.00	0.07
Cr ₂ O ₃	0.02	0.03	0.00	0.03	0.03	0.05	0.03	0.07
Total	100.21	99.36	91.24	99.91	92.76	99.82	97.32	98.42

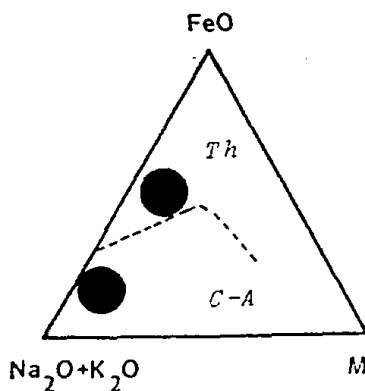


Fig. 3

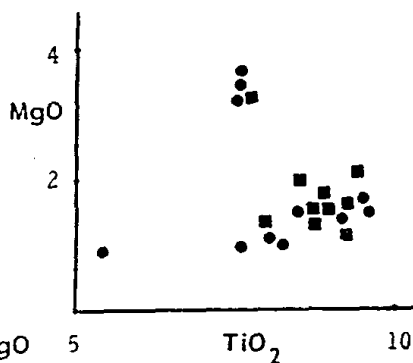


Fig. 4

Fig. 3. A-F-M diagram representing two types of volcanic glass. (Th: tholeiitic field; C. A.: calc-alkaline field.)

Fig. 4. Comparative positions of magnetites in a MgO-TiO₂ diagram. Solid squares: present study, Guadeloupe insular margin sands; solid circles: magnetites from dacites, southern Basse Terre (Guadeloupe, after Blanc, 1983).

by Maury (1984). However, the distribution and variation of volcanic glass with time and space needs further confirmation. 3. As adopted by Keller (1981), a compositional comparison of magnetites studied, with those from Basse Terre (Guadeloupe) reported by Blanc (1983), indicates that magnetites occupy the same position on a MgO-TiO₂ diagram (Fig. 4).

These observations suggest that a probable source of pyroclastic sands from the insular margins is the island of Guadeloupe. Volcanic detritus generated by island arcs is ultimately deposited in the adjacent margins and basins (Sigurdsson and Carey, 1981). Active volcanoes produce episodic eruptions, contributing to the direct deposition of aerially transported volcanic material. Such recurring phenomena result in the accumulation and preservation of pyroclastic material intercalated within marine sediments. The exact mode of transport (aerial or underwater) with respect to morphostructural features of the insular margin forms a part of ongoing investigations which are underway.

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