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Petrophysical and Microhardness Characterisation of the Sans Souci Formation, Trinidad

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Abstract: *Since 1907, the Sans Souci Formation, the only igneous outcrop in Trinidad, has been investigated using scientific procedures such as lithology, stratigraphy, petrology, paleomagnetism, microscopic analysis and mineralogy. The approach taken in this work revolved around mineralogical, topographical, elemental and microhardness distribution characteristics using various laboratory analysis techniques, including x-ray diffraction (XRD), scanning electron microscopy, physical properties characterisation and vickers hardness methods. This is an attempt at quantitatively characterising the mineralogy as well as determining the elemental distribution within the rocks of the Formation. The results are expected to contribute to existing knowledge with respect to the petrophysical and microhardness characterisation of the Sans Souci Formation. Qualitative and quantitative XRD analyses of three (3) outcrop samples studied established that the minerals calcite, chlorite and albite featured predominantly in the Sans Souci Formation. Physical characteristics such as apparent porosity, bulk density and water absorption were also determined and their values ranged 0.91-1.97%, 0.58-1.29% and 1.50-1.69g cm^{-3}, respectively. The average Vickers Hardness value for sample SS3 was determined to be 833.9.*

Keywords: *Petrophysics; microhardness; mineralogy; Sans Souci; Scanning Electron Microscopy*

1. Introduction

Trinidad is the southernmost of the West Indian islands. The Sans Souci Formation is located in the eastern region of the Northern Range and is the only igneous outcrop found in Trinidad (Cruz et al., 2007; Donovan, 1994). As early as 1907 this small outcrop, formerly known as the Sans Souci Volcanic Formation, was described in terms of lithology, geology, petrology, mineralogy and microscopy. Cunningham-Craig (1907) classified it as an epidiorite sill. Waring (1926) used microscopic analysis to establish that the igneous rocks were a combination of basaltic tuff, granophyre and diabase. Maxwell (1948) employed the same technique as Waring (1926) and obtained similar results, in that the igneous rocks were a stock of andesitic composition. Barr (1963) described the rocks as dense, lithified, dark-green to grey-green, generally fine grained and poorly sorted pyroclastic material. Wadge and Macdonald (1985) discontinued the use of the name Sans Souci Volcanic Formation and formally used Sans Souci Formation after they determined the major and trace elements and petrophysically described the chemical alteration of the rocks. Frey, Saunders and Schwander (1988) used x-ray powder diffraction to determine the mineralogy of the rock sample, but only qualitatively.

In terms of a petrophysical description of the Sans Souci Formation, the works cited above represent the most significant researches thus far. In this present study, a non-invasive laboratory approach which involved a combination of petrophysical techniques to characterise the rock samples was adopted. These included x-ray diffraction (XRD), scanning electron microscopy (SEM), physical characterisation and vickers hardness methods. With the application of these techniques, these outcrop samples can be qualitatively and quantitatively characterised in terms of their mineralogy and elemental composition as well as microstructurally defined. This work serves to augment the paucity of research done with respect to the petrophysical and microhardness characterisation of the Sans Souci Formation.

Therefore, the main objective of this research was to give a more detailed petrophysical characterisation of the igneous rocks of the Formation using a combination of advanced analytical techniques now available.

2. Materials and Methods

2.1 Sampling Technique

Samples were taken at the type section of the outcrop close to the Old Sans Souci Fishing Depot (see Figure 1).

Fresh unweathered samples were preferred in order to avoid probable misleading results should weathered samples be used. The samples were chiselled out of the rock mass beneath the regolith layer.

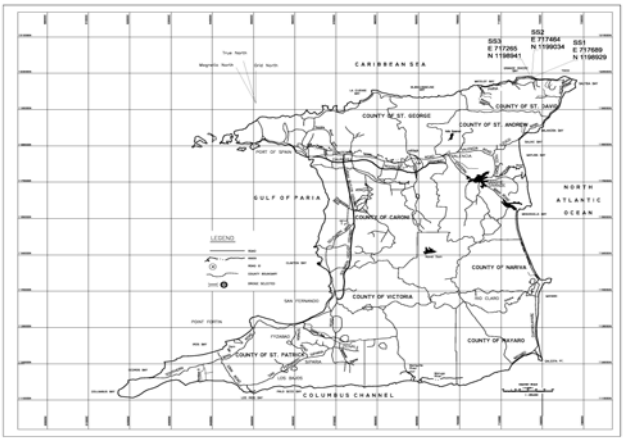


Figure 1. Sampling points for the three samples taken from the Sans Souci Formation, Trinidad

2.2 Laboratory Methods

X-ray powder diffraction

XRD and mineralogical characterisation of the rock samples was done using Cu Kα radiation in a Bruker 5005D x-ray diffractometer. Samples from the bulk rocks were ground to a particle size of less than 500 µm, and analysed over the 2-theta range 3º to 40º (Jenkins and Snyder, 1996; Knorr and Bruker, 2008; Pecharsky and Zavalij, 2009; Szponder and Trybalski, 2010). In conjunction with the qualitative profiles obtained, the TOPAS software based on the Rietveld quantitative phase analysis was used to generate quantitative data. This is a widely used and reliable quantitative phase analysis technique widely employed in both research and industrial purposes (Chalmers, Ross and Bustin, 2012; University of Cambridge, 2012).

Scanning electron microscope

Fractured sections from the bulk rocks were gold coated and imaged in a Philips 500 electron microscope operated at 20 kV. Energy Dispersive X-ray Analysis (EDXA) elemental mapping was also done using the microscope together with the EDAX Genesis elemental analyser (Goldstein et al., 2003).

Physical characterisation methods

The water displacement method based on the Archimedes principle was used to determine the apparent porosity, bulk density and water absorption of the samples (Yavuz, Demirdag and Caran 2010). The method involved obtaining a dry weight W_d, and a wet weight W_w after the open pores were saturated with water under vacuum. Subsequently, a suspended weight W_s was also obtained (ASTM, 2006); Siegesmund and Durrast, 2011). These weights were used to calculate the various physical property parameters according to:

$$\text{Apparent Porosity} = \frac{W_w - W_d}{W_w - W_s} \times 100\% \quad (1)$$

$$\text{Water Absorption} = \frac{W_w - W_d}{W_d} \times 100\% \quad (2), \text{ and}$$

$$\text{Bulk Density} = \frac{W_d}{W_w - W_s}\ gcm^{-3} \quad (3)$$

In each sample twelve (12) replicates were used to calculate the standard deviation.

Vicker's Hardness

Samples from the Formation studied in this research were set in resin, ground and polished, then mounted and tested in the Buehler, IndentaMet 1100 Series MicroIndentation Hardness Testers. This equipment was used to determine the Vickers Hardness Values for the rock samples. A load of 300 grams was used and a dwell time of 8 seconds was applied to the resin set samples.

All three samples taken from the Sans Souci Formation were prepared to determine the Vickers Hardness, however SS1 and SS2 samples could not be used due to the poor nature of the samples such that no determinations of Vickers Hardness could have been made. In addition to obtaining the hardness number, the indentation response of the sample was observed and imaged using the camera of the instrument interfaced with a computer.

3. Discussions

Table 1 shows the general description of the Sans Souci samples analysed. Generally, all three samples were similar in appearance, with the exception that SS2 displayed areas of purple and magenta hues.

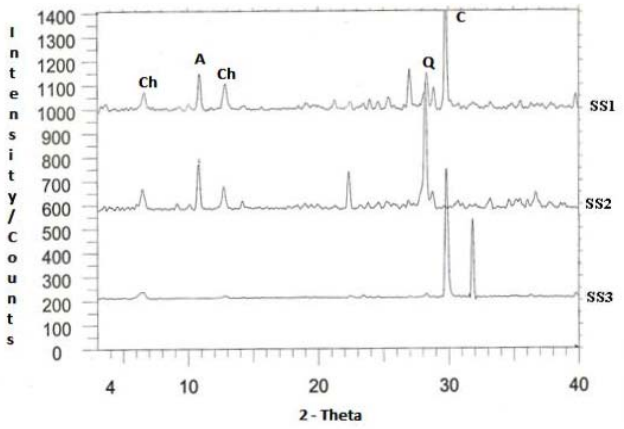
Table 1. General description of the rock samples analysed

Rock Classification	Sample	General Description
Metabasalt	SS1	Black, dark grey, angular and very dense in appearance and texture
Metagabbro	SS2	Black, grey, dark brown, purple/ magenta appearance, angular and very dense
Metabasalt	SS3	Black, grey, angular and very dense in appearance

On X-ray diffraction

Figure 2 shows normalised XRD profiles for SS1, SS2 and SS3. As can be seen, quartz, chlorite, calcite and albite were the most prominent minerals detected in the SS1and SS2 samples. In the SS3 sample, chlorite and calcite predominated. Quantitatively, Table 2 reveals that in fact SS3 was comprised mostly of calcite and chlorite and to a lesser extent albite. On the other hand, the constituents of SS1 and SS2 were mostly calcite, chlorite and albite. Qualitatively, these minerals detected in the SS1, SS2 and SS3 samples were basically the same

reported by Frey, Saunders and Schwander (1988) for their sampling site in the Sans Souci Formation.



Ch =Chlorite, **A** =Albite, **Q** =Quartz, **C** =Calcite

Figure 2. Normalised XRD profiles for samples SS1, SS2 and SS3

On scanning electron microscopy

Table 2 shows the quantitative mineralogical values obtained for rock samples. Figure 3(a) shows a SEM micrograph of a selected area of horizontally stacked, bedded intermixed calcite/chlorite/albite permeated by transverse quartz veins in the SS1 sample in which the combined calcite/chlorite content amounted to some 84%.

Table 2. The quantitative mineralogical values obtained for rock samples

Sample	Quartz %	Calcite %	Chlorite %	Albite %
SS1	3.79	63.12	20.63	12.46
SS2	4.11	17.08	31.78	47.02
SS3	0.48	76.05	14.69	8.78

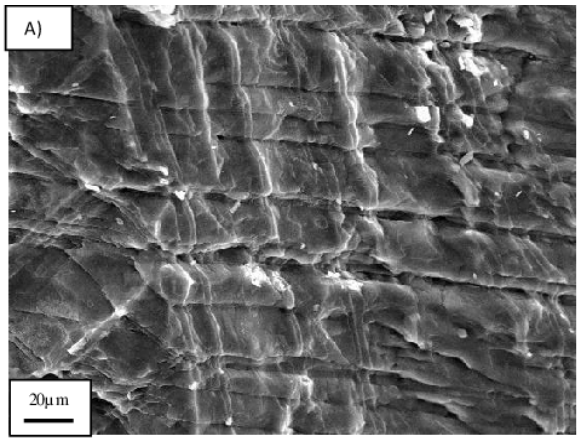


Figure 3(a). A SEM micrograph of a selected area of the SS1 sample

Higher magnification imaging in the vicinity of the quartz veins (Figure 3(b)), suggests that there may be some degree of vertical layering in the calcite/chlorite/ albite intermix. In yet another selected region of the same sample (Figure 3(c)), angular quartz grains (left) can be seen embedded in the intermix.

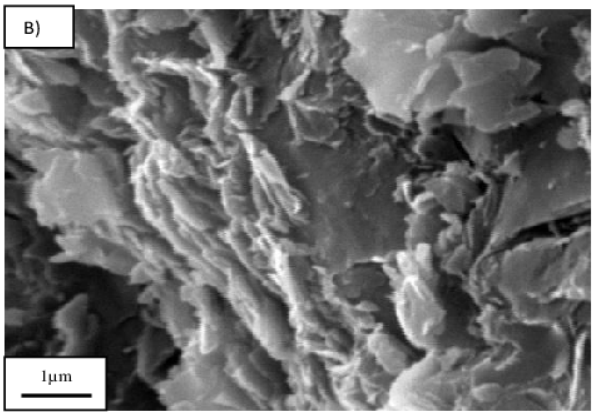


Figure 3(b). Higher magnification imaging in the vicinity of the quartz veins

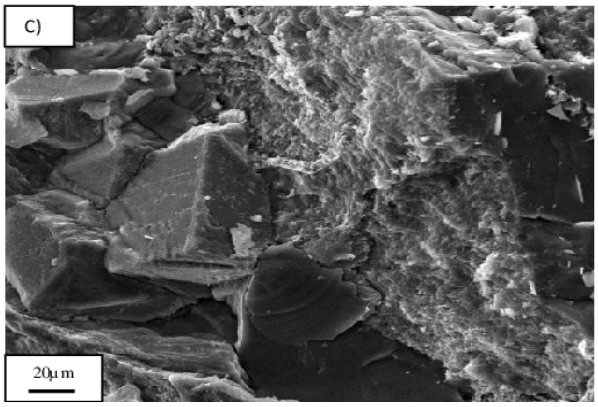


Figure 3(c). Higher magnification imaging in the vicinity of the angular quartz grains

To highlight the variability of the microstructure of the samples, Figure 4(a) shows a selected area of fine particulate calcite in the sample SS3. High magnification imaging (Figure 4(b)) further reveals a honeycomb-like texture to the particles.

Figure 5 shows a selected region of the SS1 sample with the corresponding elemental maps shown in Figure 6. Consistent with quartz, calcite, chlorite and albite being the major constituents (see Table 2), areas rich in, for example Al (Figure 6(a)), Si (Figure 6(b)) and Ca (Figure 6(d)) are clearly identifiable. Further, in addition to Fe and Ti, trace amounts of Na, V, Mn, Co, B were also detected and confirmed on the basis of the EDXA spectrum (see Figure 7) obtained from the same field of view of the sample.

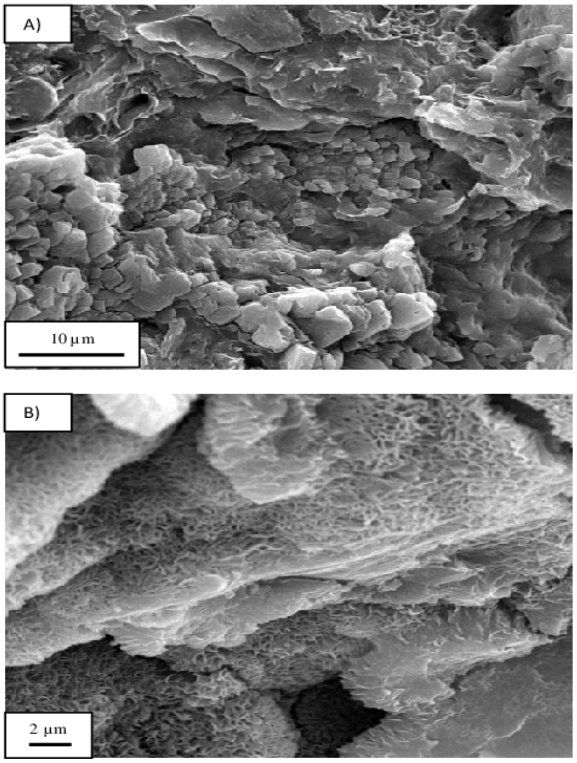


Figure 4. SEM micrograph of a selected area of the SS3 sample: (b) high magnification image of the particles seen in (a)

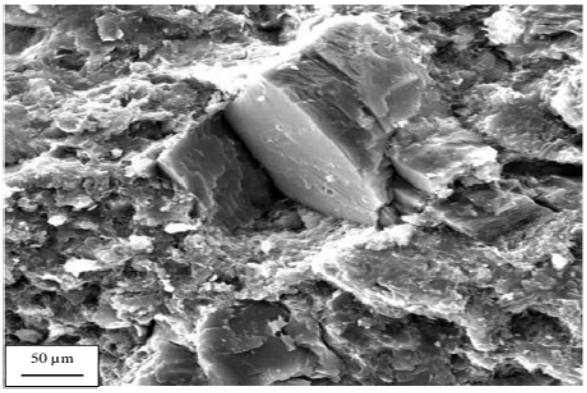


Figure 5. A SEM showing the field view of the SS1 sample from which the elemental maps and the EDXA spectrum were generated

On Physical Properties

Table 3 shows the apparent porosity, bulk density and water absorption of the samples. For all of the samples the apparent porosity and the water absorption were low.

Table 3. Physical properties of the samples

Sample Name	Apparent Porosity/ %	Bulk Density/ g cm^{-3}	Water Absorption/ %
SS1	1.44 ± 0.39	1.56 ± 0.01	0.92 ± 0.25
SS2	1.97 ± 0.46	1.69 ± 0.17	1.29 ± 0.30
SS3	0.91 ± 0.23	1.50 ± 0.10	0.58 ± 0.15

Figures 3(a)-(c) show the dense nature of the sample which agrees with the low values determined for apparent porosity and water absorption.

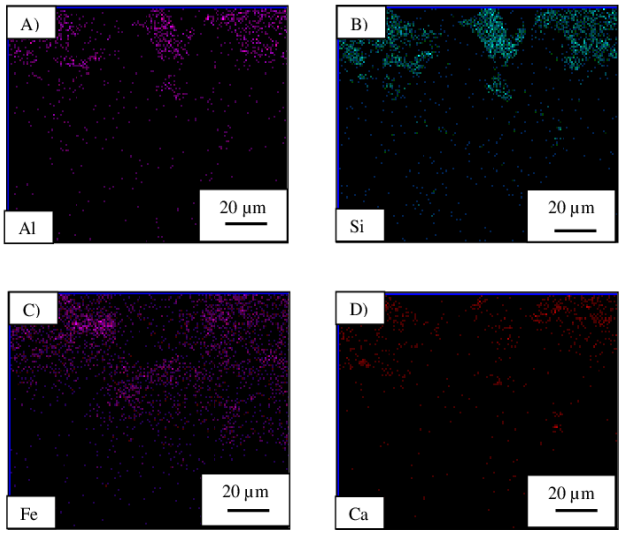


Figure 6. (a) - (d). Significant EDXA elemental maps obtained for Sample SS1

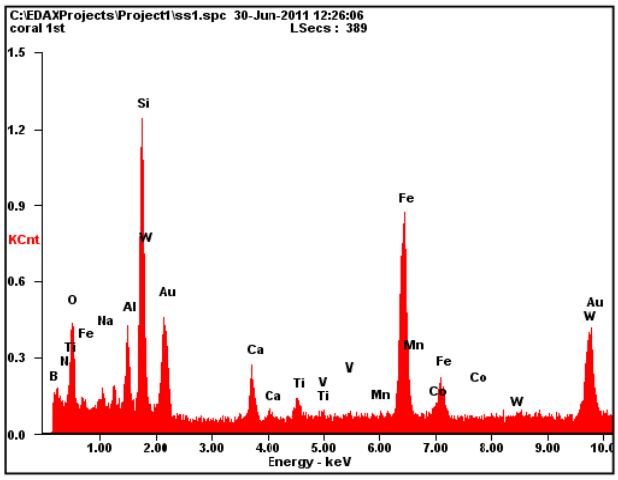


Figure 7. EDXA Spectrum obtained for sample SS1.
Note: Au derived from coating used during preparation

Vicker's Hardness

From the results shown in Table 4, the average Vickers Hardness value for sample SS3, was determined to be 833.9. The response of the rock to indentation is shown in Figure 8. Lateral cracks and lateral chipping can be seen around the indentation. Due to the indentation, the areas around the indentation appear to be pushed upwards, indicated by shiny appearances around the square based pyramid indent. However, this behaviour seen for the SS3 samples relates to that of a typical brittle material.

Table 4. The Vickers Hardness Values obtained for rock sample, SS3

Trial	Vickers Hardness Value
1	819.8
2	990.4
3	766.0
4	798.2
5	795.2

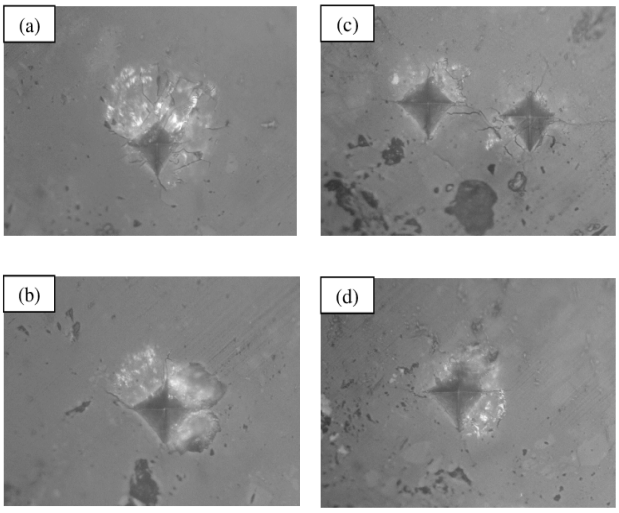


Figure 8.(a) - (d). Typical indentation response of Sample SS3

4. Conclusions

The Sans Souci Formation was characterised petrophysically/ microstructurally defined using a combination of analytical techniques which included X-Ray Diffraction, Scanning Electron Microscopy, Physical Characterisation and Vickers Hardness Methods. Physically, the rocks of the Formation are hard and dense and of low porosity with, at a microscopic level, considerable variability in microstructure.

The results of the mineralogical analysis for the type section studied, revealed that the rocks are comprised principally of calcite, chlorite, quartz and albite. Spatially, however, there is, in terms of composition, a high degree of variability to their relative proportions. The empirical hardness value determined for this Formation was relative to the micro hardness scale. The Sans Souci Formation is now microstructurally characterised. The originality of this work lies therein the analytical techniques used to microstructurally characterise the Sans Souci Formation, and the properties analysed can be further used for civil engineering purposes.

References:

ASTM (2006), *ASTM Standard C373 – 88: 2006: Standard Test Method for Water Absorption, Bulk Density,Apparent Porosity and Apparent Specific Gravity of Fired Whiteware Products*, ASTM International, West Conshohocken, PA.

Barr, K.W. (1963), "The geology of the Toco district, Trinidad, West Indies", *Overseas Geology and Mineral Resources*, Vol.8, No.4, pp.379-415.

Chalmers, G.R.L., Ross, D.J.K. and Bustin, R.M. (2012), "Geological controls on matrix permeability of Devonian Gas Shales in the Horn River and Liard Basins, Northeastern British Columbia, Canada", *International Journal of Coal Geology*: Vol.103, pp.120-131.

Cruz, L., Fayon, A., Teyssier, C and Weber, J. (2007), "Exhumation and deformation processes in transpressional orogens: The Venezuelan Paria Peninsula, SE Caribbean-South American plate boundary", *The Geological Society of America*, Vol.434, pp.149-165

Cunningham-Craig, E.H. (1907), "Metamorphic Rocks of Trinidad", Council Paper No. 76. The Government Printery, Port of Spain, Trinidad and Tobago.

Donovan, S.K. (1994), "Trinidad", In: Donovan, S. K. and Jackson, T.A. (eds), *Caribbean Geology: An Introduction*, University of the West Indies Publishers' Association, Kingston**,** p.209-228.

Frey, M., Saunders, J. and Schwander, H. (1988), "The mineralogy and metamorphic geology of low-grade metasediments, Northern Range, Trinidad", *Journal of Geological Society of London*, Vol.145, pp. 563-575

Goldstein, J., Newbury, D., Joy, D., Lyman, C., Echlin, P., Lifshin, E., Sawyer, L. and Michael, J. (2003), *Scanning Electron Microscopy and X-ray Microanalysis*, Springer, New York.

Jenkins, R. and Snyder, R.L. (1996), "Introduction to X-ray powder diffraction", *Chemical Analysis*, Vol.138, No. 1, Wiley Interscience.

Knorr, K. and Bruker, A.X.S. (2008), *Quantitative XRD Phase analysis in Minerals & Mining: Bauxite Lab.*, Report XRD 63, 1-4.

Maxwell, J. (1948), "Geology of Tobago, British West Indies", *Bulletin of the Geological Society of America*, Vol.59, pp.801-854

Pecharsky, V. and Zavalij, P. (2009), *Fundamentals of Powder Diffraction and Structural Characterisation of Materials*, 2nd Edition, Springer Science + Business Media, LLC, New York.

Siegesmund, S. and Durrast, H. (2011). "Physical and mechanical properties of rocks", In: Siegesmund, S. and Rolf Snethlage (Eds), *Stone in Architecture*, 4th Edition, Springer Science + Business Media, LLC, New York.

Szponder, D. and Trybalski, K. (2010), "Determination of progressive research methodology of using modern measuring devices to determine physical, chemical and mineralogical properties of raw materials and mineral wastes", *Physicochemical Problems of Mineral Processing*, Vol.46, pp.191-206.

University of Cambridge (2012), *X-Ray Diffractometry Facilities*, available at: http://www.esc.cam.ac.uk/resources/facilities/equipment-and-instruments/xrd/x-ray-diffractometry-software. (Accessed December 9, 2012)

Wadge, G. and Macdonald R. (1985), "Cretaceous tholeiites of the northern continental margin of South America: the Sans Souci Formation of Trinidad", *Journal of Geological Society of London*, Vol.142, pp.297-308

Waring, G. (1926), *The Geology of the Island of Trinidad, B. W. I.,* The Johns Hopkins Press, Baltimore.

Yavuz, H.S., Demirdag, S. and Caran, S. (2010), "Thermal effect on the physical properties of carbonate rocks", *International Journal of Rock Mechanics and Mining Sciences*, Vol.47, No.1, pp.94-103.

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Ricardo Marcus Clarke *is a lecturer in Environmental Physics. He holds a BSc, MPhil and PhD from The University of the West Indies. His main research area is Renewable energy, particularly focusing on solar energy. Dr. Clarke leads research at the Environmental Physics Laboratory and some of the areas include wind energy assessment, climate and climate impact analyses and petrology.*

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