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# *Research article* **Scanning Electron Microscopy (SEM) in the surficial cover analysis of the low fluvial-marine terraces in the southern coast of São Paulo state, Brazil**

*Microscopia eletrônica de varredurana análise de coberturas superficiais de baixos terraços flúviomarinhosno litoral sul do estado de São Paulo*

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**Abstract:** The Ribeira de Iguape estuarine system in Southeastern Brazil has resulted from the complex interactions between allogenic (climate, tectonics, and human interventions) and autogenic geomorphological processes. Early studies have shown that the relationship between sedimentary balances, fluvial discharge, and Holocene climate pulses resulted in the coastline displacement and low fluvial-marine terraces morphogenesis, which are composed of the marine and fluvial sedimentary units. In this perspective, this study aimed to investigate whether the microtextural and morphological records on the quartz grains associated with the different allostratigraphic units (marine and fluvial) correspond to the paleoenvironments identified in previous studies. In this study we have used a combination of analyses (grain-size, microtextural, and absolute dating) applied to the surficial cover sampled from two fluvial-marine terraces located on the right and left banks of the Iguape river channel. The results show correspondences between paleoenvironments and microtextures found on the quartz grains, confirming the statements addressed in previous studies that indicated the coastline was positioned about 9 km inland around 3.5-2.7 ka.

**Keywords:** Transgressions and Regressions; Climate Pulses; Late Holocene.

**Resumo:** O sistema estuarino do rio Ribeira de Iguape, é resultado de complexas interações entre processos geomorfológicos alogênicos (clima, tectônica e intervenções antrópicas) e autogênicos. Estudos recentes mostram que o inter-relacionamento entre balanço sedimentar, descarga fluvial e pulsos climáticos holocênicos resultou no deslocamento da linha de costa e morfogênese de baixos terraços flúviomarinhos, os quais são compostos por coberturas superficiais relacionadas a unidades marinhas e fluviais. Nessa perspectiva, este estudo teve como objetivo investigar se os registros microtexturais e morfológicos dos grãos de quartzo associados às diferentes unidades aloestratigráficas (marinhas e fluviais), apresentam correspondências com os paleoambientes deposicionais identificados em estudos anteriores. A investigação utilizou-se da combinação entre análises granulométricas, microtexturais e datações absolutas por luminescência opticamente estimulada de coberturas superficiais amostradas em dois depósitos flúviomarinhos, localizados na margem direita e margem esquerda do canal do rio Ribeira de Iguape. Por meio dos resultados, foi possível verificar correspondências entre os paleoambientes deposicionais marinhos e fluviais com as microtexturas impressas nas superfícies e grãos de quartzo. Os resultados corroboram com estudos prévios, os quais mostraram que a linha de costa já esteve localizada mais no interior do continente (~ 9 km de sua posição atual) entre 3.5 e 2.7 ka.

**Palavras-chave:** Transgressões e Regressões; Pulsos Climáticos; Holoceno Superior.

## **1. Introduction**

Scanning Electron Microscopy (SEM) has been used in Geosciences since the 1970s (e.g. Krinsley and Doornkamp, 1973; Margolis and Krinsley, 1974) and it is a useful tool in deeper investigations of the complex depositional history of geomorphological systems (Mahaney, 2002; Vos et al., 2014; Machado et al., 2016). According to Vos et al. (2014), the advantages of using SEM when compared to conventional approaches that employ optical stereo microscopy arise from the fact that information about sedimentary cycles and depositional environments can be better identified from the microtextures on quartz grains corresponding to chemical and mechanical processes over their sedimentary history. Thus, the analyses combining microtexture with other surficial cover sedimentological aspects can support depositional paleoenvironments interpretations.

The relief of the Ribeira de Iguape estuarine system (RIES), located in Southern São Paulo state, Brazil, has resulted from complex interactions between multiple geomorphological attributes (Martinez et al., 2018; Souza and Perez Filho, 2020) and the complexity associated with the area evolution also correlates with multi-scale processes such as the relative sea level (RSL) changes and marine transgression and regression episodes (e.g. Suguio et al., 1978; 1985; Angulo et al., 2006; Sawakuchi et al., 2008; Guedes et al., 2011a; Castro et al., 2014; Souza and Perez Filho, 2019). Furthermore, Souza and Perez Filho (2019) pointed out that marine transgression and regression events that occurred in the Iguape and Ilha Comprida region during the Late Holocene were triggered by oscillation on the sedimentary continent-ocean balance induced by precipitation variations established with Holocene climate Pulses. In turn, these climate events present important global-scale teleconnections despite uncorrelated environment responses between the hemispheres (BOND ET AL., 2001; BRADLEY et al., 2003; COHEN et al., 2005; CRUZ et al., 2009; PESSENDA et al., 2012; LÜNING et al., 2019).

Based on this, Souza and Perez Filho (2019) identified different levels of the fluvial-marine terraces in RIES showing morphostratigraphic records of the Late Holocene marine transgressions and regressions. According to the authors, the depositional package associated with the low fluvial-marine terraces on the right bank of the river presents important erosive-depositional discontinuities related to the marine and fluvial allostratigraphic units which indicate distinguished depositional processes. The depositional history of these surficial covers may also be recorded in the microtextures on quartz grains found therein.

From this perspective, the aim of this research was to investigate if the quartz grain microtextures and morphologies from the fluvial-marine deposits correspond with the depositional environments previously identified by Souza and Perez Filho (2019). Additionally, the surficial cover of the other hypothesized fluvial-marine terrace on the left bank that presents unclear erosive-depositional discontinuities, contrasting with that located on the right bank of the Ribeira de Iguape river, was also analyzed. The assumption is that the microtextures on the quartz grain surfaces record processes associated to the different depositional environments established during the marine transgression and regression events, contributing to the identification of discontinuities resulting from marine and fluvial depositional paleoenvironments.

## **2. Study area**

The study area is an estuary-type geomorphological system characterized as long, narrow, partially closed channels that extend across an alluvial plain and which are connected to the open sea (Perillo, 1995; Hugget, 2007; Montagna et al., 2013). The RIES comprises the lower part of the homonymous river - named Lower Ribeira de Iguape river (LRIR) - located in the Southern littoral of São Paulo state and partially encompassing Sete Barras, Miracatu, Juquiá, Registro, Pariquera-Açu, Iguape, and Ilha Comprida municipalities (Figure 1). The river mouth is inserted in the Iguape-Cananéia lagoon complex, which is formed by the Comprida, Iguape and Cananeia islands, and also by the fluvial-lagoon system named "Mar Pequeno" to where important regional fluvial systems flow.

*Revista Brasileira de Geomorfologia. 2022, v.23, n. 1; (Jan-Mar)* DOI: 10.20502/rbg.v23i1.1959 http://www.lsie.unb.br/rbg/ In the study area it is possible to identify a lowland morphostructural unit named "Tectonic Depression of the Low Ribeira", which is subdivided into three morphoesculptural sub-units: the Tectonic Depression of the Lower Iguape River, the Cananéia-Iguape Coastal Plain, and the Fluvial Terraces and Plains of the Lower Iguape River (Ross, 2002). In Figure 1b it is possible to observe the lowered topography ranging from the 0 to 40 m a.s.l where the samplings were conducted, which contrasts with an altitude exceeding 1,000 m close to Sete Barras, Juquiá and Registro municipalities. According to Sousa et al. (1996), the lowered sector is inserted in the "Iguape Valley" whose genesis is associated with the Guapiara shear zone (NW-SE) neotectonic reactivation and where the slopes rarely reach  $10^\circ$ , contrasting with the crystalline sectors where the slope angles exceed  $70^\circ$ .

Additionally, the landforms associated with the highest altimetry and slope values are supported by granites, mylonites, batholiths, and schists with Proterozoic ages (Peixoto and Theodorovictz, 2009). Mesozoic alkaline intrusive rocks are also present (Jacupiranga Alkaline Complex and Juquiá Alkaline Complex); they are associated with the hills that stand out near the mouth of the Ribeira de Iguape river. In the "Iguape Valley" and close to the coastal plain, sedimentary strata integrating the Pariquera-Açu and Cananéia formations can be found. These rocks are from the Cenozoic era and are correlated to the N50-N60 Sequence that occurs in the Santos Sedimentary Basin (Moreira et al., 2007).



**Figure 1**. Location of the RIES. A) Localization in the São Paulo state. B) Altimetry. Yellow spots: sample sites. TFM-MD: low fluvial-marine terrace on the right bank. TFM-ME: low fluvial-marine terrace on the left bank.

This area is inserted in the Cananeia Seismogenic Zone (IPT, 1984; Mioto, 1993), where the lineaments are E-W, NE-SW, and NW-SE oriented following the shear systems associated with the Cubatão Mega-Fault, such as the Guapiara Lineament (Sadowski, 1991). Heilbron et al. (1995; 2004) verified the important influence of the Ribeira Mobile Belt - a Brasiliano Orogen system - on the regional geomorphological development. According to Souza and Perez Filho (2020), this structural framework acted as a background to the development of the Iguape River Mainstem and its affluents during the Late Holocene.

These geological and geomorphological settings in the LRIR led to the development of the meandering fluvial channel and surficial cover depositions whose texture varies from the silty to clayey, which are found especially close to urban areas of the Registro and Iguape municipalities. Melo (1990) identified surficial cover in association with sandy-clayey alluvial soils sand-clay alluvium, gravels, talus deposits, strand-plains, mangrove deposits, sandy sediments (current beach), and undifferentiated mixed-deposition sediments in the coastal plain. Additionally, IAC (1999) pointed out soils of the order of cambisols, latosols, argisols, spodosols (Brazilian classification).

#### **3. Materials and Methods**

#### *3.1. Fluvial-marine identifications and erosive-depositional discontinuities into the deposits*

The different levels of low terraces were identified by using field surveys, satellite images, and SRTM v.2 Digital Elevation Model (DEM). The DEM used height correction trough exclusion of spurious pixels and the subsequent filling of no-data pixels using the natural neighbor interpolating method, while the low fluvial-marine terrace on the left bank was identified and analyzed following Souza and Perez Filho (2019), which previously identified the low fluvial-marine terrace on the right bank.

The discontinuities into the deposits were identified based on analyses of morphological aspects, colors (Munsell Color Chart), sedimentary and pedogenetic features present in the fluvial-marine deposits (e.g. Bigarella and Mousinho, 1965; Meis, 1977; Miall, 1990; Corrêa, 2001; Daley and Cohen, 2018). Later, the grain size analyses and optically stimulated luminescence dating also contributed to the identification and interpretation of the discontinuities. In the depositional packages depictions, it is easy to observe aspects concerning the texture and the sedimentary and pedological structures of the surficial covers (e.g. ferruginous concretion, mottling, cerosity, stratification, bioclasts, organic-matter laminations and minerals observable in macroscopic scale). Furthermore, colors similar to those from the Munsell Color System (1994) were used in the depictions.

In this way, the labels summarize and identify the low terrace type ("Fm" for fluvial-marine); the location of the deposit ("MD" for right bank and "ME" for left bank of the Ribeira de Iguape); depositional process ("f" for fluvial, "m" for marine and "l" for lagoon); and the stacking sequences from top to bottom ("a" to "e"). The aim was to identify major discordances recording changes in the erosive-depositional processes following the methodological approach pointed out by Souza and Perez Filho (2019).

#### *3.2. Grain size analyses*

Seven surficial covers sampled from low terraces on the right and left banks (5 samples from the right bank and 2 samples from the left bank) of the Ribeira de Iguape river were analyzed. The sampling was performed from base to top of the deposits, avoiding cross contaminations and preventing interference in the grain-size results and microtextural analyses.

Later, each of the seven 100g aliquots was split into very coarse sand (1.00 mm / 0 $\phi$ ), coarse sand (0.50 mm / 1.5ϕ), medium sand (0.25mm / 2 ϕ) and fine sand (0.125 mm / 3 ϕ), very fine sand (0.053 mm / 4.05 ϕ), silt (0.002 mm  $/9 \phi$ ) and clay (0.0002 mm, 10  $\phi$ ) fractions. The technical procedures involved the use of sieves with openings in the aforementioned diameters and the use of a pipette for separation of the clay and silt fractions (Camargo et al., 2009). The values obtained after the aliquots fractionation were plotted on ternary diagrams (Flemming, 2000), and the grain-size parameters (i.e. kurtosis, skewness, and sorting) were also calculated according to the method of moment (Swan et al. 1978, 1979). The GRADISTAT v.8 was used to calculate the parameter values (Blott and Pye, 2001) and the results were presented in the phi scale.

The grain size results from the right bank presented by Souza and Perez Filho (2019) were used in this study. They correspond to the samplings conducted at 0-20, 30-40, 55-70, 10-105, and 105-120 cm depth. The samplings we conducted on the left bank were done at 0-20 and 30-40 cm depth. In both studies, due to the limited financial resources, the procedure sought to optimize the available resources considering the spatial distribution of the low fluvial-marine terraces and the most expressive erosive-depositional discontinuities found into the depositional packages. Therefore, the grain-size analyses aimed to identify the major discontinuities into the deposits that may indicate changes in depositional processes throughout the Late Holocene.

#### *3.3. Scanning Electron Microscopy (SEM)*

The microtexture analyses using SEM were conducted on quartz grain with sizes varying between 250 $\mu$ m (0.25 mm) and 1000µm (1.00 mm) were randomly selected from the same surficial covers samples used in the grain size analyses. The grains were selected using a polarized light microscopy (PLM) available in the Mineral Quantification Laboratory located in the Institute of Geoscience from the State University of Campinas (UNICAMP).

During the preparation for the SEM analyses there was no need to submit the quartz grains to the chemical cleaning procedures pointed out by Vos et al. (2014) since during the fractionationing steps the samples had

already been subjected to clay minerals and organic matter dispersion procedures. The selected quartz grains were allocated in the specific samplers, metallized with carbon and analyzed using a Secondary Electron Detector coupled to a Scanning Electron Microscope (SEM). Finally, the interpretations about the depositional environments associated with the found microtextures were supported by Vos et al. (2014).

## *3.4. Optically Stimulated Luminescence (OSL)*

OSL dating was performed on the 4 surficial cover samples (3 on the right bank and 1 on the left bank of the Ribeira de Iguape river), which were collected in different depths and avoiding truncations between erosive-depositional discontinuities. Dark PVC tubes with 15 cm in diameter and 50 cm in length at 35, 65, and 95 cm depths on the right bank and at 35 cm depths on the left bank were used for the samplings. Later, the samples were sent to the "Laboratório de Datação LTDA" to obtain the absolute ages following Huntley et al. (1985), Cordier et al. (2010), Murray and Wintle (2000) and Nelson et al. (2015) and applying the SAR (Single Aliquot Regenerative-Dose) protocol to 15 aliquots (MURRAY; WINTLE, 2000).

## **4. Results**

#### *4.1. Site samples*

The samplings were conducted in the low fluvial-marine terraces identified on the Ribeira de Iguape river right and left banks. On the right bank, the sample site corresponds to the T2 terrace level that is positioned about 9 km away from the current intertidal zone, approximately 5 meters above the RSL ( $\sim$ 2 m above river channel water surface) and presenting slopes that do not exceed 3º (Souza and Perez Filho, 2019; Figure 2a). In Figure 2a, other two surfaces positioned at 7.5 and 15 m a.s.l. were identified, showing the occurrence of another low terrace level (level T1) and a third low terrace level that was imprecisely delimited due to DEM resolution (30 m) and difficulties in accessing that sector of the landscape.



**Figure 2**. Cross-section of the low fluvial-marine terraces on the Ribeira de Iguape river. A) Low fluvial-marine on the right bank. B) Low fluvial-marine on the left bank. TFM-MD: sample site corresponding to the low fluvial-marine terrace on the right bank. TFM-ME: sample site corresponding to the low fluvial-marine terrace on the left bank. Source: Souza and Perez Filho (2019).

On the left bank, two low terraces levels were identified to  $\sim$ 2.5 and  $\sim$ 1.5 m a.s.l (Figure 2b). However, the samplings were conducted in the T2  $(-1.5 \text{ m a.s.})$  terrace which is about 100 m away from the river mainstem, to at 8.5 km away from the current intertidal zone and present slopes that do not exceed 8º. Similarly to the right bank low fluvial-marine terrace, this landform also corresponds to the Unit 3 from the Guedes et al. (2011a, b).

## *4.2. Surficial covers*

The depositional package supporting the low fluvial-marine terrace on the right bank of the Ribeira de Iguape river is 130cm thick and it presents discontinuities, which are related to three depositional processes (fluvial, marine and fluvial-lagoon; Figure 3A). Therefore, the package is organized into the 7 major discontinuities that were differentiated by grain-size, morphological, and chemical features (SOUZA; PEREZ FILHO, 2019).

According to IAC (1999), soil classes of the spodosol can be found in the sites where the surficial covers were sampled. In contrast, undifferentiated mixed-deposition sediments resulting from fluvial, marine and lacustrine depositional processes are mentioned by Melo (1990). In this work the layers into the deposits are understood as records of the different depositional processes (Souza and Perez Filho, 2019) occurring throughout the Late Holocene, for this reason Melo's (1990) proposal is considered more suitable for this work.

Between 0-20 cm depth (Fm-Md-fa), the sandy fractions (71.4%) are predominant in surficial covers especially that consistent with "fine sand" fraction that represents 64.4% of the whole sample (Figure 3b). The surficial covers are poorly sorted, very leptokurtic and very finely skewed. The Flemming's (2000) diagram shows that the samples are classified in "Muddy" (sediment type) and "Clayey Sand" (B-III; textural classification). According to Munsell Chart (1994), the surficial cover found in this depth are grayish brown (10YR 5/2) and pedogenetic features (ferruginous concretions, mottling, and cerosity). In addition, micaceous minerals likely upstream sourced from the crystalline and sedimentary rocks were also found (SOUZA; PEREZ FILHO, 2019).



**Figure 3.** Surficial cover deposit of the fluvial-marine terrace on the right bank. A) Allostratigraphic profile. B) Grain-size curves. FM: Fluvial-marine. MD: Right bank. fa: Fluvial depositional processes. ma to me: Marine depositional processes. la: fluvial-lagoon depositional processes. Source: Adapted from the Souza and Perez Filho (2019).

The surficial covers at 30-40 cm depth (FM-MD-ma) has the predominance of sandy fractions (92.0%), mainly in relation to "medium sand" fraction (68.4%) (Figure 3b). The calculation of the grain-size parameters indicated that the sample is moderately well sorted, very leptokurtic and coarse skewed, while the Flemming's (2000) diagram shown the sample is classified as "Muddy"(sediment type) and "Slightly Clayey Sand" (A-II; textural classification). Additionally, plane-parallel stratification, bioclasts in millimeter sizes and carbonate nodules with dissolution pores were found in this layer. Finally, according to Munsell Chart (1994) the sample is light gray  $(10\text{Y}R\ 7/1)$  and the OSL dating was  $3.24\pm0.51$  ka.

The surficial cover sampled at 55-70 cm depth (FM-Md-md) has predominance of the sandy fractions (86%), but presents a subtle increase in the fine fractions than the above layers (Figure 3b). According to Souza and Perez Filho (2019), this may be a result from finer materials translocation from the topper layers and indicate an incipient illuviation process. The grain-size parameter calculation has indicated the sample is moderately well

sorted, very leptokurtic, and finely skewed. According to Flemming's (2000) diagram, the sample can be classified as Slightly Muddy Sand (sediment type) and Slightly Silty Sand (A-I; textural classification). Also, plane-parallels stratification, bioclasts and carbonate nodules were found. In addition, the color in accordance with Munsell Chart (1994) is "weak red" (2.5 YR 7/6) and the OSL absolute dating was  $3.4\pm0.6$  ka.

At 70-50 cm depth (FM-Md-me) are predominant sandy fractions (96.4%) (Figure 3b) and the grain size parameters calculation indicated the sample is moderately well sorted, very leptokurtic, and very coarse skewed. The hydrodynamics classification indicated the "sand" (S) classes for both the sediment type and the textural class. According to Munsell Chart (1994) the sample color is white (2.5 YR 8/1) and the OSL absolute dating was 3.57±0.59 ka.

Finally, at 105-120 cm depth (FM-Md-la) the results also indicated in the sample the sandy fractions are predominant (94.5%) (Figure 3b). Consequently, the calculation of the grain-size parameters indicated the sample is moderately well sorted, very coarsely skewed, and very leptokurtic. In addition, the Flamming's (2000) diagram shown that the sample is classified as slightly muddy sand (sediment type) and slightly silty sand (A-I; textural classification), while the Munsell Chart (1994) indicated the light olive brown color (2.5 YR 5/6) which is consistent with field observation indicating organic-matter and sulphides concentration. In turn, these features indicate the establishment of low hydrodynamic energy depositional environments (fluvial-lagoon environment).

The depositional package related with the low terrace on the left bank has 70 cm thickness and gradual transitions between the discontinuities (Figure 4b). This deposit is closer to the current fluvial channel, resulting in a more efficient connectivity between frequent floods and weathering on the depositional package. Further, the mottling from at 50 cm depth toward the deepest layer suggests the groundwater table is close to the surface. Therefore, the coupling between both superficial and sub-superficial weathering may be resulted in a more gradual transitions between the 4 discontinuities associated with marine (FM-Me-ma, FM-Me-mb, FM-Me-mc) and fluvial (FM-Me-fa) depositional processes found into the deposits.



**Figure 4.** Surficial cover deposit of the fluvial-marine terrace on the left bank. A) Allostratigraphic profile. B) Grain-size curves. FM: Fluvial-marine. Me: left bank. fa: Fluvial depositional processes. ma to me: Marine depositional processes.

The 0-20 cm depth (FM-Me-fa) sample has fine fractions predominance (52.5%), mainly concerning the silt fraction (32.6%). The calculation of grain-size parameters indicated the sample are poorly sorted, very leptokurtic, and very finely skewed whereas the Flemming's (2000) diagram shown "sandy mud" (sediment type) and "silty sand mud" (C-III; textural classification) classes. These results suggest the depositions occurring in low energy environments, similarly to the fluvial allostratigraphic unit on the right bank. The Munsell Chart (1994) shown gray color (10YR 6/1).

The FM-Me-ma discontinuity has brown color (10YR 5/3) and sandy fractions increments were observed during the field surveys, because the laboratory analyses were performed only in the most expressive discontinuities considering the financial support constraints. On the other hand, the results at 30-40 cm depths (FM-Me-mb) have shown sandy fraction predominance (92.1%) wherein the "fine sand" fraction is predominant (59.2%). The calculation of the grain size parameters has shown the sample is moderately sorted, leptokurtic, and finely skewed. According to Munsell Chart (1994), the surficial cover is light gray color (10YR 7/2) and the Flemming's (2000) diagram indicated the slightly muddy sand (sediment type) and slightly silty sand (A-I; textural classification) classes. Plane-parallel stratification was observed and the OSL dating was 3.0±0.46 ka, similarly to the depositional marine unit into the fluvial-marine deposit on the right bank. Finally, the results suggest depositions in a more energetic environment than verified to the FM-Me-fa discontinuity

The FM-Me-mc discontinuity is also light gray (10YR 7/2) and mottling was found. Although laboratory analyses were also not performed in this layer, during the field surveys a sandier texture was observed similarly to the FM-Me-mb discontinuity.

#### *4.3. Quartz grain microtextures*

The analyzed samples collected at 0-20 cm depth (Fm-Md-fa) from the right-bank low terraces shown quartz grain with bulbous edges, solution pits, v–shaped percussion cracks, conchoidal fractures presenting arcuate e straight steps on the planes, chattermarks enlarged by dissolutions, scratches e crescentic percussion marks (Figure 5). According to Vos et al. (2014), these microtextures indicate the grains suffered fluvial transport followed by chemical alterations and are consistent with the grain size analyses and statement presented by Souza and Perez Filho (2019), which pointed out the establishment of the low energy depositional environments linked to fluvial processes.



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**Figure 5.** Microtextures on quartz-grains of surficial covers sampled at 0-20 cm depth (FM-Md-fa) in the low fluvial-marinho terrace on the Ribeira de Iguape river right bank, São Paulo state. A) Angular and subangular predominant morphologies on quartz grains. B) Angular quartz grain. a) straight steps. b) chattermarks. c) adhering particles. d) oriented etch pits. e) solution pits. C) Sub-rounded quartz grain with chemical dissolutions and v-shaped percussion cracks microtextures. D) Silica precipitation on the upturned plates. E) Quartz grains sub-rounded morphology and mechanical marks resulting from fluvial transport processes. F) Focus on the microtextures shown in the "E". a) v–shaped percussion cracks. b) chattermarks. c) chemical dissolutions microtextures. d) straight scratches.

The analyzed quartz grain of the surficial cover sampled at 30-40 cm depth (Fm-Md-ma) has crescentic percussion marks, chattermarks, v–shaped percussion cracks, straight scratches, bulbous edges, oriented etch pits (triangle), silica globules/flowers, straight steps, conchoidal fractures, and flat cleavage surfaces (Figure 6). Vos et al. (2014) indicated the "flat cleavage surfaces" are associated with aeolian transports, suggesting the investigated surficial cover may have been reworked by the wind when the coastline was located more inland than currently (Souza and Perez Filho, 2019). Also, the aforementioned authors pointed out that the silica globules/flowers/pellicles indicate environments with low energy where conditions to silica precipitation on quartz grain are created. On the other hand, frequently these microtextures are also found on quartz from the intertidal zone as a result of the successive submersion and exposure in silica saturated environments.



**Figure 6.** Quartz grain microtextures from the surficial cover sampled at 30-40 cm depth (FM-Md-ma) in the low fluvial-marine on the right bank of the Ribeira de Iguape river, São paulo state. A) Angular and subangular quartz grain. B) Sub-rounded quartz grain. a) v–shaped percussion cracks. b) Straight steps. C) Sub-rounded grains with bulbous edges. D) Microtextures. a) Oriented etch pits. b) Silica flowers. c) v–shaped percussion cracks. E) Rounded quartz grain. F) Microtextures on the quartz grain of the image "E". a) flat cleavage surfaces. b) Straight steps. c) chattermarks. d) Solution pits.

The results of microtexture analysis on the quartz grains from the surficial cover sampled at 55-75 cm depth (FM-Md-md) shown medium conchoidal fractures (~50µm), v–shaped percussion cracks, straight scratches, chattermarks, high reliefs, solution pits, silica globules e flat cleavage surfaces (Figure 7). Additionally, on the quartz grain concerning to surficial cover sampled at 75-105 cm (Fm-Md-me) were identified upturned plates, curved grooves, v–shaped percussion cracks, solution pits, silica globules/flowers, adhering particles, conchoidal fractures, and straight steps (Figure 8). Following Vos et al. (2014), these microtextures are indicating depositional processes associated with two environments: aeolian and marine subaqueous.



**Figure 7.** Microtextures on quartz grains from the surficial cover sampled at 55-70 cm depth (FM-Me-md) of the low fluvial-marine terrace on the left bank of the Ribeira de Iguape river, São Paulo, state. A) Predominance of the angular and sub-rounded morphologies. B) Sub-rounded quartz grain with bulbous edges. C) Focus on the microtextures in the "B" image. a) chattermarks. b) v–shaped percussion cracks. c) crescentic percussion marks. D) Sub-angular quartz grains. a) conchoidal fractures. b) Straight scratches e straight steps. c) Dissolution marks. E) Upturned plates and chemical alterations. F) Microtextures on the conchoidal fractures. a) conchoidal fractures. b) percussion marks. c) Straight steps and straight scratches. c) Multiples v–shaped percussion cracks.



**Figure 8.** Microtextures on quartz grains from the surficial cover sampled at 70-105 cm depth (FM-Md-me) in the low fluvial-marine terrace on the right bank of the Ribeira de Iguape river, São Paulo state. A) Predominance of the sub-rounded morphologies in the sample. B) Sub-rounded grains (a), rounded grain (b), and weathering plagioclases (c). C) Straight scratches microtextures on the sub-rounded grain. (a), oriented etch pits (b), v–shaped percussion cracks (c), and small conchoidal fractures (d). D) Chemical microtextures. a) Oriented etch pits (triangles). b) Straight scratches. c) Adhering particles. E) Microtextures on the conchoidal fracture plane. a) Straight steps. b) Multiple v–shaped percussion cracks. c) Adhering particles. d) Straight e curved scratches. F) Sub-rounded grain (a) and weathered plagioclase (b), arcuate steps (c), conchoidal fractures (d), v–shaped percussion cracks (e) e adhering particles (f).

Concerning the low terrace on the left bank, the microtexture analyses were conducted on the quartz grains selected from the surficial cover sampled at 50 cm depth (FM-Me-mc). Therefore, the results have shown weathered grains with oriented pits, v–shaped percussion cracks, straight, arcuate steps, upturned plates, elongated depressions, and polygonal cracks microtextures (Figure 9). Although these grains are very chemically altered, microtextures associated to subaqueous (upturned plates, elongated depressions, and straight arcuate steps) and intertidal processes could be identified (Vos et al., 2014). Particularly about the elongated depressions, Vos et al. (2014) affirm its association with polygenic processes (chemical and mechanical) resulting from the aeolian transport. For this work, these features reinforce deposition processes in the coastal environments.



**Figure 9.** Microtextures on quartz grains from the surficial cover sampled at 30-50 cm depth (FM-Me-mb) in the low fluvial-marine terrace on the left bank of the Ribeira de Iguape river, São Paulo state. A) Predominance in the sample of the weathered angular and sub-angular quartz grain. B) Angular quartz grain with polygonal crack elongated by chemical alterations. C) Sub-rounded quartz grain. a) Elongated depressions. b) Oriented etch pits. c) Straight scratches. d) Silica flowers. D) Focus on the microtexture in the "C" image. a) Oriented etch pits. b) v–shape percussion cracks. E) Sub-angular quartz grain. a) Chemical dissolution marks. b) arcuate steps. F) Arcuate steps microtexture. a) Mineral inclusion record. b) Chemically altered upturned plates.

## **5. Discussions**

The fluvial-marine terraces are constituted of surficial cover resulting from the land surfaces and oceanic erosive-depositional processes. Therefore, these landforms are important geomorphological records in the coastal landscapes of the paleoenvironments. In the area, Souza e Perez Filho (2019) indicated that the low fluvial-marine geneses is inherently connected with the Holocene Climate Pulses (HCP) triggered by orbital, oceanographical, atmospheric processes (e.g. O'BRIEN et al., 1995; HAUG et al., 2001; MOY et al., 2002; WANNER et al. 2011; CRUZ et al., 2009).

From this, Ljungqvist (2010) pointed out that in last 2,000 years at least 4 temperature change events have been verified in Europe: Roman Warm Period (1-300 C.E.), Dark Age Cold Period (300-800 C.E.), Medieval Warm Period (800-1.300 C.E.) e Little Ice Age (1.300-1.850 C.E.). Further these main climate events, some studies have also identified increases in the frequencies of the low magnitude climate events since 5.0 ka (e.g. Hauget al., 2001). Additionally, the aforementioned paleoclimate dynamics are correlated with the Holocene sedimentary history in the RIES where low fluvial-marine terraces are recording coastline displacements from at 3.5 ka to present (Souza e Perez Filho, 2019). Consequently, our results have shown the occurrence of depositional environments successions, which are related with different hydrodynamic conditions, presenting important correlations with previous studies.

The sandy surficial cover ranging from the well to moderately sorting and deposited between at 3.57±0.59 ka and 3.24±0.51 on the right bank of the RIR (FM-Md-ma à FM-Md-me), has microtextures consistent with the marine depositional environments established when the coastline was positioned farther inland Souza and Perez Filho, 2019; Souza et al., 2020). Therefore, was verified on the right bank quartz grain with the predominance of the morphologies varying from the sub-angular to angular further the microtextures associated with the mechanical processes suggesting sub-aqueous transports in the intertidal environments. Usually, in this kind of environment, relatively well hydrodynamic energy occurs (Vos et al., 2014). Conchoidal fractures sized < 50µm, straight steps, flat cleavage surfaces, straight scratches, upturned plates, crescentic percussion marks, bulbous edges, and curved grooves are mentioned by the literature as originated by frictions between different grain surfaces resulting in breakages during the sub-aqueous transport.

Additionally, the combined chemical and mechanical processes microtextures (oriented etch triangles, chattermarks, silica globules, silica flowers e adhering particles) suggest the surficial covers related to the FM-Md-ma, FM-Md-mb, FM-Md-mc, FM-Md-md, and FM-Md-me units remained under aqueous and silica saturated environments after depositions. Therefore, the genesis of silica globules/flowers on the quartz from the FM-Me-md is associated with constant grains immersion and submersion in fluid supersaturated by silica (Higgs, 1979) likely influenced by the intertidal variations. Consequently, the marine units related with the low fluvial-marine on the right bank may have been a former marine-built terrace whose morphogenesis is associated with upward sediment accretions linked to intertidal variations. This hypothesis can be endorsed considering the plane-parallel stratifications found into the marine units of the fluvial-marine deposit on the right bank (SOUZA; PEREZ FILHO, 2019).

Thus, the discontinuities in the marine units of the RIR right bank deposit seem to have resulted from the upper layers fine translocation as pointed out by Souza and Perez Filho (2019). Also, grain size results indicating fine fractions increases in the FM-Me-md discontinuity and the adhering particle on the quartz grains reinforces the illuviation processes in layers differentiation process. In addition, the high weathered quartz grain of the FM-Md-me layer and mottling presence in deeper layers indicate the significant influence of the groundwater table oscillation acting on the grains chemical alteration.

According to Bull et al. (1981) the chattermarks results of the chemical alteration after grain collision, while Vos et al. (2014) affirm that although there is no specific environment for this microtexture they have also been reported as originated from coastal processes. In addition, the bulbous edges resulting from the aeolian transports reinforces that the marine unit depositions were by coastal process. Therefore, coupling these results with grain size analyses that shown sandy fractions predominance, well sorting, and A-I classification (Flemming, 2000) surficial covers was made it possible identify the intertidal depositional environments establishment between 3.57±0.59 ka e 3.24±0.51 ka.

The surficial cover from marine units into the fluvial-marine deposit on the left bank (FM-Me-mb), has quartz grain with very similar microtextures to that found in the marine units of the deposits on the right bank. However, microtextures resulting from high chemical alterations and consistent with field observation were identified. In turn, in the field survey were identified mottling and gradual transition between the different erosive-depositional discontinuities indicating intense activity of the chemical weathering. On the other hand, in the fluvial-marine package on the right bank the sedimentary structures that report different depositional processes are most well preserved. The low fluvial-marine terrace on the left bank is positioned closest to the fluvial channel and contrasts with the low terrace on the left bank which is located near an oxbow lake, making its depositional package "disconnected" from the river. Likely these settings contributed to a more weathering efficiency in the deposit chemical alteration due to the water availability.

Although the mechanical microtextures are chemically very alterated, we identify that its transports occurred in association with high hydrodynamic energy sub-aqueous environments linked to the coastal area. Moreover, the elongated depressions microtexture indicates aeolian transport (Mahaney, 2002) whereas straight scratches are indicative of the coastal depositional environments (Vos et al., 2014). In this way, our results suggest a mixing depositional processes (intertidal and aeolian) to the surficial cover related with the FM-Me-Mb discontinuity and that subsequently suffered strong weathering due to establishment of the low energy environment establishment in consonance with the coastline retreat in the Late Holocene.

These statements are also supported by grain size results showing the sample is moderately sorted, very finely skewed and A-I classification (Flemming, 2000), similarly to marine units into the fluvial-marine deposit on the right bank. Also, the depositional age 3.0 ±0.46 ka is consistent with the low terrace on the right bank confirming depositions in the coastal environments and identification of the paleo-coastline. Consequently, the OSL ages differences verified between the marine units from both the low fluvial marine terraces located to approximately at 9km (right bank) and 8,5 km (left bank) away from the current coastline suggests relationships with the marine regressions started about the 2.7 ka (PESSENDA et al., 2012; SOUZA;PEREZ FILHO, 2019; SOUZA et. al, 2020).

Therefore, during the coastline regression progressively the coastal depositional environments were replaced by the fluvial environment resulting in the depositions of the FM-Md-fa and FM-Me-fa discontinuities over the marine units (Souza and Perez Filho, 2019; Souza et al., 2020). In the FM-Md-fa unit, the results indicated association with a lower hydrodynamic energy, contrasting with the surficial covers related to the marine units. Furthermore, the grain size characterization of the fluvial units of both the banks indicating the surficial covers are poorly sorted and Flemming's (2000) diagram varying from B-III (FM-Md-fa) to C-III (FM-Me-fa) classes reinforces this hypothesis. Additionally, the analysis of the quartz grain from the FM-Md-fa have shown predominances of sub-angular and sub-rounded morphologies suggesting that they were transported over long distances in the low-energy fluvial processes. According to Mahaney (2002), the gains transported in the low-energy fluvial flux varies from the sub-angular to angular morphologies further presenting a certain degree of rounding as a result of the water transport.

Contributing to the discussions, v-shaped percussion cracks, straight steps, chattermarks, and conchoidal fractures microtextures indicating grain collisions in underwater environments were identified (Vos et al., 2014) further chemical dissolution marks that suggest the establishment of the low energy environments. Moreover, Souza and Perez Filho (2019) showed that pedogenetic features in the fluvial layers into the fluvial-marine deposits are common suggesting a high intensity of the weathering altering theses surficial cover in both the banks.

Our results corroborate the results of previous studies and contribute new information for a more detailed paleoenvironmental interpretation on the geomorphological dynamics of the RIES during the late Holocene. Moreover, these results reinforce the issues addressed in this work discussing the depositional processes and geomorphological dynamics coupled to the coastline retreat about the 2.7 ka further point out that multiproxies approaches are necessary in studies on this thematic.

The multiproxy approach when applied to similar studies developed in humid regions is a very useful tool, since the humid tropics conduct an intense chemical weathering that removes the microtextures that could record depositional environments. However, while microtextural analyzes enable the result refinement of studies on surficial covers, they also have limitations which arise from the sedimentary history and pedogenetic transformation of the deposit. Therefore, a combination of methodologies can lead to more reliable interpretations. For instance, Machado (2016) identified depositional paleoenvironments in the Espírito Santo state littoral using several sedimentological approaches that included microtextural analyses.

The microtexture analyses on the quartz grain from the surficial covers associated with fluvial-marine located on both banks of the LRIR has shown consistent results with the depositional processes and environments previously identified, increasing the paleoenvironmental interpretations of the study area. In this context, the previous identification of the erosive-depositional discontinuities into the low fluvial-marine terrace on the right bank subsidized the assumptions about the paleoenvironments linked to the low terraces on the left bank and allowed more accuracy in the interpretation of the results.

## **6. Conclusions**

Scanning Electron Microscopy (SEM), through the secondary electron detector, was a useful tool for the study of the surficial covers related to low fluvial-marine terraces. The application of this methodology enabled the identification of the relationships between different paleoenvironmental resulting from coastline displacements during the Late Holocene. Consequently, we can assess the combination of different methodologies that included optically stimulated luminescence dating in studies on coastline evolution in humid Tropical environments.

Therefore, the correspondences between marine depositional environments at about 3.5 ka and the microtextures on the quartz grain contributed to identify intertidal depositional environments and improved the previous paleoenvironmental interpretations. Moreover, based on the microtextures, we can confirm that the fluvial allostratigraphic units were deposited by fluvial processes during the marine regressions from at 2.7 ka. Nonetheless, the geochronological correlations between both low terraces resulting from the characterization of the fluvial-marine deposits on the left bank have shown the coastline was located approximately to 8.5 km away from the current position at about 3.0 ka.

Finally, the microtexture analyses are useful methodologies to obtain more precise results and interpretations on the geomorphological dynamic in the continent-ocean transitional areas during the Late Holocene climate pulses. The study of the climate pulse fingerprints requires contemporary analytical approaches which can subsidize results with high resolutions, reinforcing the importance of multiproxy studies about the geomorphological processes and dynamics on the coastal areas.

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