

# *Pesquisas em Geociências*

<http://seer.ufrgs.br/PesquisasemGeociencias>

---

**Depositional age definition of the Açú Formation  
(Potiguar Basin, northeastern Brazil) through <sup>40</sup>Ar-<sup>39</sup>Ar dating of  
early-auriferous K-feldspar overgrowths**

*Anderson Maraschin, Ana Maria Mizusaki, Paulo Vasconcelos,*

*Ruth Hinrichs, Luiz De Ros, Sylvania dos Anjos*

*Pesquisas em Geociências*, 37 (2): 85-96, maio/ago., 2010.

Versão online disponível em:

<http://seer.ufrgs.br/PesquisasemGeociencias/article/view/22649>

---

Publicado por

**Instituto de Geociências**

---



**Portal de Periódicos**  
**UFRGS**

UNIVERSIDADE FEDERAL  
DO RIO GRANDE DO SUL

---

## **Informações Adicionais**

**Email:** [pesquisas@ufrgs.br](mailto:pesquisas@ufrgs.br)

**Políticas:** <http://seer.ufrgs.br/PesquisasemGeociencias/about/editorialPolicies#openAccessPolicy>

**Submissão:** <http://seer.ufrgs.br/PesquisasemGeociencias/about/submissions#onlineSubmissions>

**Diretrizes:** <http://seer.ufrgs.br/PesquisasemGeociencias/about/submissions#authorGuidelines>

---

Data de publicação - maio/ago., 2010.

Instituto de Geociências, Universidade Federal do Rio Grande do Sul, Porto Alegre, RS, Brasil

## Depositional age definition of the Açu Formation (Potiguar Basin, northeastern Brazil) through $^{40}\text{Ar}$ - $^{39}\text{Ar}$ dating of early-authigenic K-feldspar overgrowths

Anderson J. MARASCHIN<sup>1</sup>, Ana Maria MIZUSAKI<sup>2</sup>, Paulo M. VASCONCELOS<sup>3</sup>, Ruth HINRICHES<sup>2</sup>, Luiz F. DE ROS<sup>2</sup> & Sylvia M. C. dos ANJOS<sup>4</sup>

1. CEPAC - Centro de Excelência em Pesquisa sobre Armazenamento de Carbono, Pontifícia Universidade Católica do Rio Grande do Sul - Av. Ipiranga, 6681 - Porto Alegre - RS, Brasil, CEP: 90619-900. E-mail: anderson.maraschin@pucrs.br

2. Instituto de Geociências, Universidade Federal do Rio Grande do Sul, Av. Bento Gonçalves, 9500, Porto Alegre - RS, Brasil. E-mails: ana.mizusaki@ufrgs.br; lfderos@inf.ufrgs.br; ruth.hinrichs@ufrgs.br

3. Department of Earth Sciences, University of Queensland, Brisbane, Qld 4072, Australia. E-mail: vasconcelos@uq.edu.au

4. PETROBRAS - E & P - EXP/ST/MSP, Av. Chile, 65, Rio de Janeiro, RJ, Brasil. E-mail: anjos@petrobras.com.br

Recebido em 08/2009. Aceito para publicação em 08/2010.

Versão online publicada em 24/12/2010 ([www.pesquisasemgeociencias.ufrgs.br](http://www.pesquisasemgeociencias.ufrgs.br))

**Abstract** - Early-authigenic K-feldspar overgrowths are widespread in the Cretaceous fluvial Açu Formation sandstones from the Potiguar Basin, one of the most important onshore oil reservoirs of Brazil. These overgrowths were formed at near-surface conditions (early eodiagenesis), directly on detrital K-feldspar grains. The physical continuity between overgrowths and K-feldspar grains rendered impossible their separation by conventional means for  $^{40}\text{K}$ - $^{40}\text{Ar}$  isotope analysis. An in situ  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  step-heating method was, therefore, applied and the minimum age of 120 Ma obtained for the authigenic overgrowths agrees with the depositional biostratigraphic age previously estimated for the Açu Formation. The method applied to the Açu Formation sandstones shows excellent potential for depositional age determination of other reservoir-sandstones. We suggest that future developments of the method will, however, greatly benefit from the use of ultra-violet (UV) laser for reducing chances of contamination of authigenic K-feldspar overgrowths analyses by detrital grains.

**Keywords:**  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  dating, K-feldspar overgrowths, reservoir-sandstones, Potiguar Basin.

**Resumo** - DEFINIÇÃO DA IDADE DEPOSICIONAL DA FORMAÇÃO AÇU (BACIA POTIGUAR, NORDESTE DO BRASIL) ATRAVÉS DA DATAÇÃO  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  DE CRESCIMENTOS AUTIGÊNICOS PRECOSES DE K-FELDSPATO. Crescimentos autigênicos precoces de K-feldspato são abundantes nos arenitos fluviais cretácicos da Formação Açu (Bacia Potiguar), um dos mais importantes reservatórios *onshore* de hidrocarbonetos do Brasil. Estes crescimentos foram formados diretamente ao redor de K-feldspatos detríticos, em condições superficiais (eodiagenese inicial). A continuidade física entre crescimentos e grãos detríticos impossibilitou sua separação visando a aplicação do método convencional  $^{40}\text{K}$ - $^{40}\text{Ar}$ . Assim, optou-se em aplicar o método  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  diretamente nos crescimentos autigênicos de K-feldspato. Como resultado, foi obtida uma idade mínima de 120 Ma, muito próxima à idade bioestratigráfica previamente estabelecida para os arenitos da Formação Açu, o que demonstra a viabilidade da aplicação do método  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  na determinação de idades deposicionais de arenitos-reservatório. Entretanto, para fins de aprimoramento do método e melhor precisão nas idades, é sugerido o uso de equipamentos que utilizam sonda a laser ultravioleta (UV), o que reduz a possibilidade de contaminação por diferentes fases minerais.

**Palavras-chave:** datação  $^{40}\text{Ar}$ - $^{39}\text{Ar}$ , crescimentos autigênicos de K-feldspato, arenitos-reservatório, Bacia Potiguar.

### 1. Introduction

Sandstones represent the most important hydrocarbon reservoir rocks in many basins around the world. Nevertheless, the depositional age of several reservoir-sandstones is poorly constrained, due to the poor biostratigraphic dating. The imprecise chronostratigraphic characterization of these sandstones greatly reduces the preci-

sion of the generation-migration-accumulation models developed for their exploration and development as hydrocarbon reservoirs. Sandstones, however, contain authigenic minerals that can be used for dating (Maraschin & Mizusaki, 2008). Among the most useful are authigenic K-feldspar overgrowths precipitated around detrital K-feldspar grains during early diagenesis, soon after deposition (Sibley, 1978; Ali & Turner, 1982; Girard

*et al.*, 1988; De Ros *et al.*, 1994; Hagen *et al.*, 2001; Maraschin *et al.*, 2004; Sandler *et al.*, 2004; Baron *et al.*, 2008). The radiogenic dating of these overgrowths may provide a minimum age for the deposition of host sandstones.

An *in situ* <sup>40</sup>Ar-<sup>39</sup>Ar laser dating technique was applied to authigenic K-feldspar overgrowths in sandstones from Açú Formation in the Potiguar Basin, one of the main onshore oil reservoirs in Brazil. The fluvial sandstones of the Açú Formation are potentially suitable for <sup>40</sup>Ar-<sup>39</sup>Ar dating because contain abundant very early-authigenic K-feldspar overgrowths precipitated around detrital orthoclase and microcline grains, which constitute up to 6 percent of the whole rock. The authigenic K-feldspar overgrowths were apparently precipitated soon after deposition and before other diagenetic constituents, including mechanically infiltrated clay coatings (Maraschin *et al.*, 2004).

The radiometric dating of early-authigenic K-feldspar overgrowths offers the possibility to solve the minimum depositional age of the Açú Formation and of other clastic sequences, which biostratigraphic dating is inexistent or stratigraphic correlation is imprecise.

## 2. Geological setting

### 2.1. The Potiguar Basin

The Potiguar Basin is one of the main oil-bearing basins in Brazil covering an area of 22,000

km<sup>2</sup> onshore and 27,000 km<sup>2</sup> offshore (ANP, 2005). The basin is located in the northeastern part of the Brazilian equatorial margin (Fig. 1) related to the Gondwana break-up. The basement of this basin consists of rocks of the Borborema Province which constitutes an important paleoproterozoic tectonic unit entirely contained in the South American Platform (Almeida *et al.*, 1981; Santos & Brito Neves, 1984). According with Schobbenhaus *et al.* (1984), it is represented by sialic material reworked during the Brasiliano/Pan-African Tectonic Cycle (Late Proterozoic to Early Paleozoic), with large areas of extensively reworked, old continental crust comprising metasedimentary belts associated with granite-gneiss-migmatite terrains, in places intruded by granite batholiths (Sial, 1986).

The thick sedimentary fill of the Potiguar Basin (*ca.* 8 km thick; Fig. 2) was accumulated during the rift, transitional and drift tectonic stages of the passive margin evolution associated with the Neocomian (Late Jurassic to Early Cretaceous) South America / Africa break-up (Françolin & Szatmari, 1987; Matos, 1987; Bertani *et al.*, 1990).

The sedimentary succession is represented by the Berriasian to early Aptian Pendência Formation (Souza, 1982), which consists of lacustrine shales (source rocks), as well as sandstones and conglomerates ascribed to fluvial, deltaic and turbidite deposits. The rift phase also comprises the early Aptian Pescada Formation (Araripe & Feijó, 1994), which includes deltaic and turbiditic sandstones and shales. The rift stage ended with differential uplift and tilting of blocks, marked by a

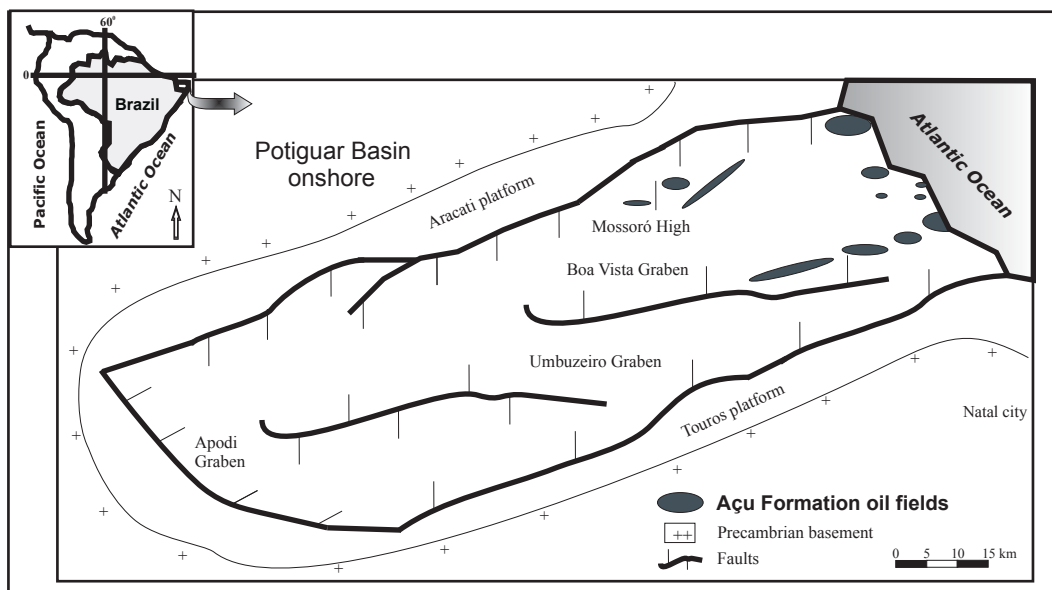


Figure 1. Location map of the Potiguar Basin (modified after Souto Filho *et al.*, 2000.).

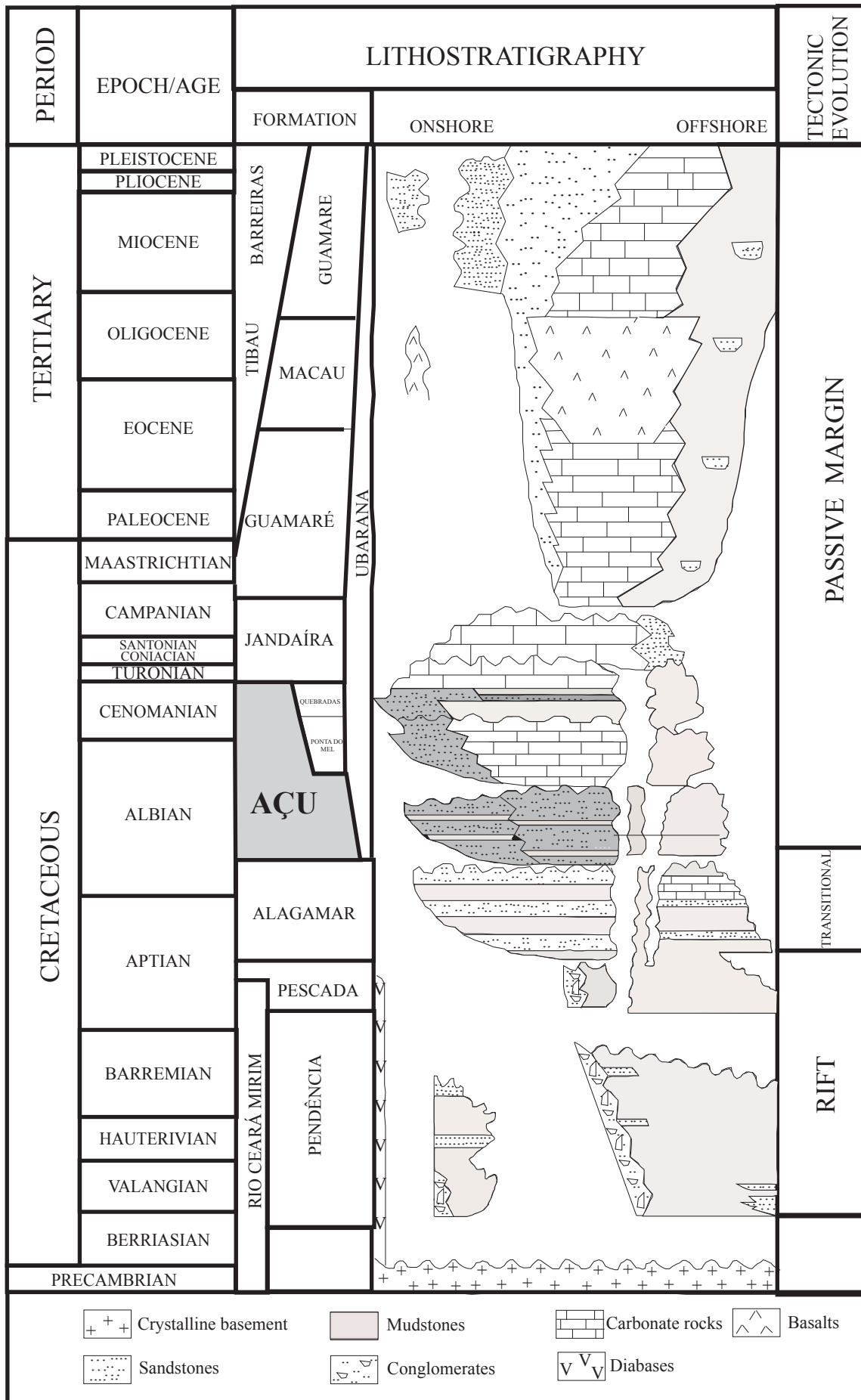


Figure 2. Stratigraphic column of the Potiguar Basin (modified from Araripe & Feijó, 1994).

widespread regional unconformity. Diabase dykes intruded the basin margins during this phase (Rio Ceará-Mirim Formation; Araripe & Feijó, 1994). Marine flooding related to the transitional stage took place during the Aptian and caused the deposition of black shales, marls and calcilitites of the Alagamar Formation. In the onshore area, this transitional section consists of the drift-stage Albian-Cenomanian fluvial-estuarine sandstones and green to red mudstones of the Açú Formation (Kreidler & Andery, 1949) that are covered by the Turonian to Campanian, shallow marine carbonates of the Jandaíra Formation (Sampaio & Schaller, 1968). In the late Cretaceous (middle Campanian), an important tectonic reactivation caused regional uplift and erosion, deforming particularly the carbonate rocks of the Jandaíra Formation (Cremonini, 1995).

During the last stage of basin evolution, a regressive sequence, comprising the Campanian to Lutetian Guamaré Formation carbonates (Souza, 1982) and the Campanian to Recent Ubarana Formation shales (Araripe & Feijó, 1994), was deposited mainly offshore. In the Eocene-Miocene, olivine-basalts of the Macau Formation were extruded. The alluvial sandstone of the Barreiras Formation, presumably of Miocene or Pliocene age constitutes the youngest sedimentary succession of the basin (Falkeinhain *et al.*, 1977).

## 2.2. The Açú Formation

The Açú Formation is one of the most important onshore hydrocarbon reservoirs among Brazilian sedimentary basins. About 70 percent of the Potiguar Basin hydrocarbon production is from these sandstones (ANP, 2005). The Açú Formation crops out along the Potiguar Basin margin (Fig. 1) and extends to the subsurface along the entire onshore portion (about 650 m thick) and down the continental shelf (around 1000 m thick), covering a total area of 40,000 km<sup>2</sup>.

The Açú Formation includes fine to very coarse sandstones with thin intercalations of shales, mudstones, siltstones, and conglomerates of alluvial, fluvial, and estuarine facies deposited during the passive margin stage of the Potiguar Basin (Fig. 2). Because of the dynamic depositional character of the formation, no lithostratigraphic markers are recognized on a regional scale. Four subunits were defined according to electric logs, identified from bottom to top as Açú 1, Açú 2, Açú 3

and, Açú 4 (Vasconcelos *et al.*, 1990). The subunits correspond to an alluvial-fan and fluvial system (Açú 1); a transgressive, fining upwards, fluvial sequence (Açú 2); an alluvial-fluvial, high-energy sequence (Açú 3); and a transgressive, coastal-estuarine system (Açú 4), which is covered by the shallow marine carbonates of the Jandaíra Formation.

Palynological analysis has suggested an Albian-Cenomanian age for the Açú Formation (Araripe & Feijó, 1994), although no precise dating is available due to the lack of worldwide correlatable fossils.

## 3. Materials and methods

One sandstone sample with abundant early-authigenic K-feldspar overgrowths was selected for <sup>40</sup>Ar-<sup>39</sup>Ar radiometric dating based on the analyses previously made by Maraschin *et al.* (2004). The authors applied different analytical methods to confirm the presence and precocity of the K-feldspar overgrowths, as petrographic examination, scanning electron microscopy, microprobe and cathodoluminescence.

The selected sample was gently disaggregated in agate mortar and placed under ultrasonic bath and running water for carbonate and clay minerals coatings removal. Afterward, the material was dried at 60° C for 2 hours. The clean sand was sieved to concentrate the 0.250 to 1 mm size range which feldspars were most abundant as determined by petrographic observation. In the next phase, K-feldspar was separated from mica and heavy minerals with a Frantz Magnetic Separator and from quartz by heavy density liquid (Bromoform 2.60 d). The residual concentrate was only detrital feldspar-rich with K-feldspar overgrowths, and minor quantities of quartz grains, no other impurities that could compromise the <sup>40</sup>Ar-<sup>39</sup>Ar analyses.

The dating of authigenic K-feldspar overgrowths by conventional <sup>40</sup>K-<sup>40</sup>Ar geochronology according to the method proposed by Lee & Savin (1985) and applied by Girard *et al.* (1988) requires the physical isolation of pure diagenetic fractions. However, owing to the lack of fluid or solid inclusions between the grains and the overgrowths of the K-feldspars of the Açú Formation, the physical separation between detrital and diagenetic phases was not achieved. Furthermore, it seemed impossi-



ble to avoid totally the contamination by detrital fragments that would have added radiogenic argon and led to erroneously older ages (i.e., ages related to rocks that originated detrital grains). Therefore, the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  method was preferred, as the release of the argon can be performed in situ by melting the overgrowths with a collimated laser beam.  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  laser dating is especially suitable for dating K-feldspar overgrowths considering Girard & Onstott (1991), Hagen *et al.* (2001), Sherlock *et al.* (2005) and Mark *et al.* (2005; 2006; 2007; 2008).

To  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  dating, 50 grains of K-feldspar with easily identified overgrowths based on their distinctive morphology (rhombic adularia habit) and dimension were selected from the concentrate through handpicking under a binocular microscope. The grains were loaded in 21-pit aluminium irradiation disks together with fraction concentrate sanidine standards, using the geometry illustrated in Vasconcelos *et al.* (2002), and irradiated for 35 hours in the IEA-R1 Reactor (Nuclear Research Institute at São Paulo University). After a 35 days cooling period, 2 K-feldspar grains were loaded into wells in a copper disk, and the disk inserted into the sample chamber in an ultra-high vacuum extraction line, and incrementally heated with a coherent Ar ion laser, operated in continuous mode with a defocused (2mm-diameter) laser beam. The fractions of gas extracted were expanded through a cold trap maintained at -138 °C, and successfully cleaned, for about 5 minutes, in a C50 SAES getter pump operated at *ca.* 450 °C and subsequently cleaned in a second getter pump, operated at 25 °C, for about 2 minutes. The cleaned gas fractions were allowed to expand into an MAP-215-50 mass spectrometer equipped with an additional C-50 SAES getter pump. The gas was ionized in the a Nier ion source and analyzed, by peak hopping, in an off-axis electron multiplier with 450 resolution and  $8.6 \times 10^{-4}$  A.torr<sup>-1</sup> abundance sensitivity. In addition to the incremental heating analysis, we also attempted to separately sample the detrital and diagenetic feldspar fractions by heating each area with a focused continuous Ar-ion laser beam. Afterwards, the 46 irradiated grains were gently crushed immersed in absolute ethanol in an agate mortar, and reloaded 146 fragments of crushed feldspar into individual wells in a 221-well copper disk. To each well, we added a small grain of zero age glass to ensure effective coupling between the blue-green Ar-ion laser beam and the white to transparent feldspar fragments. These 146 K-

feldspar grains represent a population comprising pure detrital feldspar cores, various ratios of feldspar cores with authigenic feldspar overgrowths, and pure grains of diagenetic feldspar overgrowths. These grains were fused with a focused 6-W Ar Ion laser (Coherent Innova 306) beam, and the gas released was cleaned and analyzed as described above. The objective was to obtain statistically the ages of pure detrital feldspars and of authigenic feldspars overgrowths. All these procedures were performed at the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  laboratory of the Geochronology Research Center of the Geosciences Institute at the São Paulo University.

## 4. Results

### 4.1. Detrital texture and composition of the Açú Formation sandstones

Maraschin *et al.* (2004) classified the Açú Formation sandstones as arkoses (Folk, 1968; average  $Q_{65.9}F_{32.6}L_{1.9}$ ), mostly medium- to coarse-grained, very poorly to moderately-sorted (sorting 1.2), with angular to rounded, mostly subangular to subrounded grains. The more frequent detrital constituent is quartz, with an average (av.) of 40 bulk volume %. Feldspars are abundant (av. 20%), with a predominance of microcline (av. 12%) over orthoclase (av. 7%) and minor plagioclase (av. 0.1%). The only volumetrically important rock fragments (av. 0.1%) are felsic plutonic (granitic /gneissic) in the coarsest sandstones. Mud intraclasts (av. 2%) are common in the coarse-grained sandstones, being usually compacted into pseudomatrix (av. 0.7%) and, in places, partially dissolved. Muscovite (av. 0.4%) is concentrated in the fine- to medium-grained sandstones and often extensively kaolinized. Biotite is less common (av. 0.3%). Detrital heavy minerals (av. < 0.1%) are represented by a relatively stable assemblage of rutile, garnet, zircon, staurolite and tourmaline.

### 4.2. Diagenesis

The diagenesis of the Açú Formation sandstones is characterized by a widespread and very early occurrence of K-feldspar as overgrowths around orthoclase and microcline grains. These rocks have undergone intense diagenetic alteration and the paragenetic sequence (Fig. 3) of constituents and processes was inferred from textural

relationships observed in thin section by Maraschin *et al.* (2004). The authigenic K-feldspar overgrowths were apparently precipitated soon after deposition, under the direct influence of the surface conditions with meteoric fluids and before other diagenetic constituents. The main diagenetic processes affecting the Açu Formation sandstones are: (1) meteoric eodiagenesis under warm, semiarid to sub-humid conditions, responsible for dissolution of detrital K-feldspar, followed by precipitation of authigenic K-feldspar overgrowths as the main phase, authigenic smectite rims, quartz overgrowths and kaolinite smectite/mixed-layer illite-smectite (I/S) as coatings and pore-fillings; (2) mesodiagenesis under shallow burial (approximate 2 km), marked by precipitation of calcite, dolomite, siderite, illite-smectite and pyrite; (3) telodiagenetic modifications promoted by meteoric flushing included dissolution of carbonate cements, and in places of K-feldspar grains and overgrowths, sometimes accompanied by the precipitation of kaolinite and finally hematite. The compaction and cementation had similar importance in porosity reduction, with a significant population of sandstones with very low porosity due to pervasive, pre-compactional cementation. Carbonates are the most abundant diagenetic constituents and, together with compaction and mechanical clay infiltration, are the main controls on reservoir quality (Maraschin *et al.*, 2004).

mate 2 km), marked by precipitation of calcite, dolomite, siderite, illite-smectite and pyrite; (3) telodiagenetic modifications promoted by meteoric flushing included dissolution of carbonate cements, and in places of K-feldspar grains and overgrowths, sometimes accompanied by the precipitation of kaolinite and finally hematite. The compaction and cementation had similar importance in porosity reduction, with a significant population of sandstones with very low porosity due to pervasive, pre-compactional cementation. Carbonates are the most abundant diagenetic constituents and, together with compaction and mechanical clay infiltration, are the main controls on reservoir quality (Maraschin *et al.*, 2004).

Diagenetic Process	Eodiagenesis	Mesodiagenesis	Telodiagenesis
K-feldspar dissolution	—		—
<b>K-feldspar overgrowth</b>	—		
Infiltrated clays		—	
Fe-oxides		—	—
Authigenic smectite		—	—
Quartz overgrowth		—	
Kaolinite		—	
Calcite		—	
Dolomite		—	
Siderite			—
Illite-Smectite			—
Calcite dissolution			—
Pyrite			—

Figure 3. Paragenetic sequence of constituents and processes during the diagenetic evolution of the Açu sandstones, showing K-feldspar overgrowth as main diagenetic phase (modified after Maraschin *et al.*, 2004).

### 4.3. Texture and composition of the authigenic K-feldspar overgrowths

Well-developed authigenic K-feldspar overgrowths are widespread on orthoclase and microcline grains (Fig. 4A) of the Açu Formation sandstones, constituting up to 6 % of the whole rocks. Overgrowths are optically discontinuous (epitaxial) relative to the detrital grains, untwined, non-luminescent and formed directly on the surface of the detrital K-feldspar grains. There are no solids (clays, oxides) or fluid inclusions between grains and overgrowths. Overgrowths often show a rhomboedral (adularia) habit (Baskin, 1956), which is usually developed parallel to the long axes of feldspar grains, as in others authigenic K-feldspar overgrowths observed elsewhere

(Stablein & Dapples, 1977; Waugh, 1978; Ali & Turner, 1982; Girard *et al.*, 1988; Krainer & Spötl, 1989; Milliken, 1989; Walgenwitz *et al.*, 1990; Lee & Parsons, 2003; Mark *et al.*, 2005). The large size of the overgrowths, about 0.09 to 0.2 mm width and 0.8 mm length (Fig. 4A), and their continuity around the feldspar grains suggest a very early precipitation. This is further indicated by the lack of inclusions of clay minerals or iron oxides between detrital grains and overgrowths and their covering by mechanically infiltrated clay coatings (Fig. 4B-C). Transmission electron microscopy (TEM) analyses clearly show microtextural differences between detrital and authigenic K-feldspar (Fig. 4D), as observed in Lee & Parsons (2003) study.

Electron microprobe analyses of the overgrowths (Maraschin *et al.*, 2004) reveal a nearly stoichiometric, pure end-member  $KAlSi_3O_8$ . They present an average composition of  $SiO_2$  (65.5%),  $Al_2O_3$  (18.1%) and  $K_2O$  (17.4%), only trace contents of Na (0.11 wt% NaO) and Ca (0.03 wt% CaO) and no Ba or Sr, as other diagenetic feldspars (Kastner & Siever, 1979; Ruppert, 1987; Saigal *et al.*, 1988).

The detrital K-feldspar grains display a bright blue cathodoluminescence, which is typical of high temperature feldspars (Smith & Stenstrom, 1965), whereas K-feldspar overgrowths lack cathodoluminescence, an usual attribute of authigenic K-feldspars (Kastner & Siever, 1979; Saigal *et al.*, 1988; Morad *et al.*, 1989).

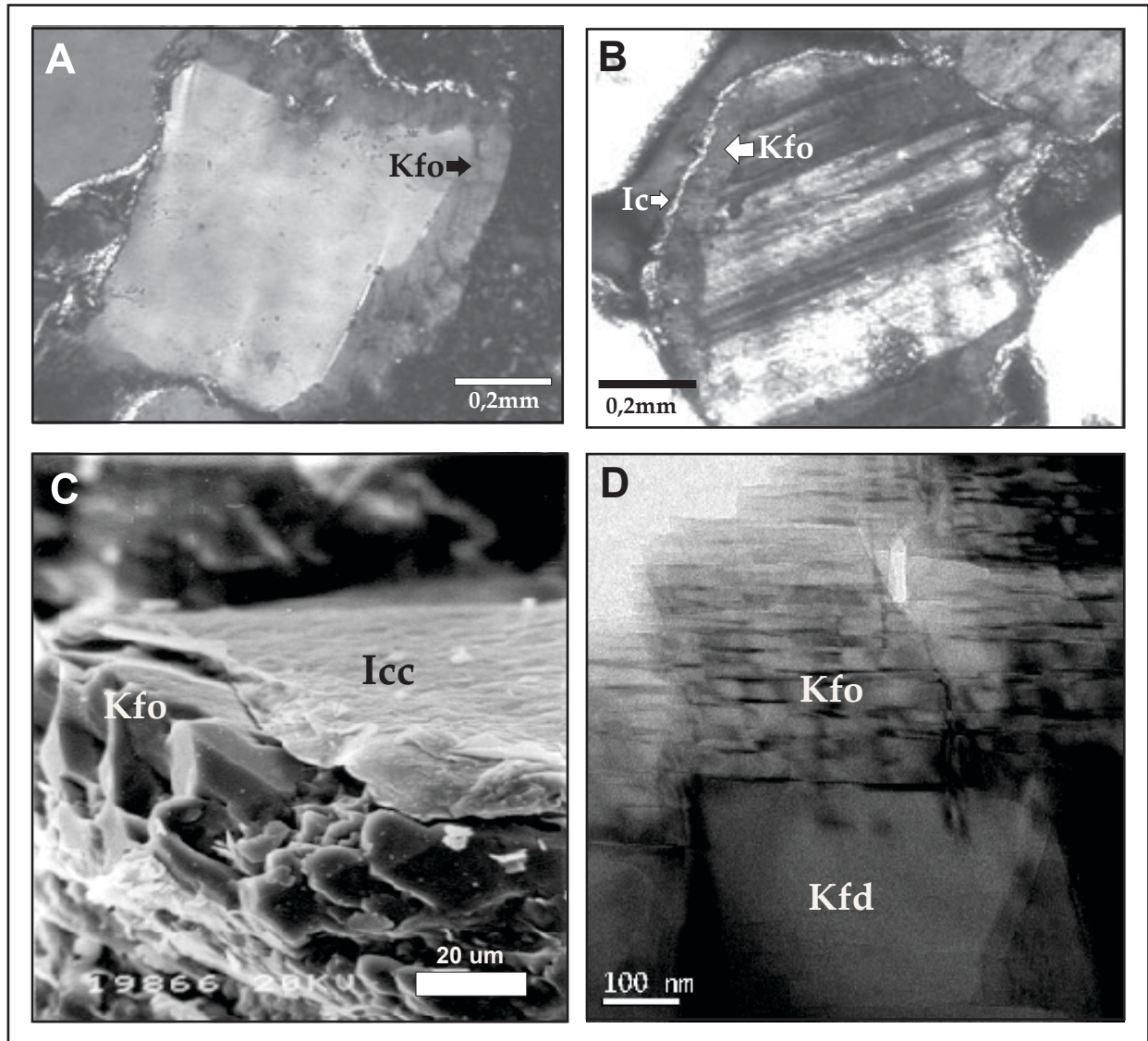


Figure 4. Cross-polarized light micrographs (A and B) and SEM image (C) Authigenic K-feldspar overgrowth (Kfo) with mechanically infiltrated clay coatings (Icc) on overgrowth (Kfo). (D) TEM image showing the microtextural differences between detrital (Kfd) and authigenic K-feldspar overgrowth (Kfo).

#### 4.4. $^{40}Ar-^{39}Ar$ analytical results

Two grains from the 50 grains previously irradiated were analyzed by the incremental step-heating method in order to extract Ar from both authigenic overgrowths and detrital K-feldspars. One of the grains (identified as 1153-01) shows a spectrum (Fig. 5) with older apparent age (about

295 Ma) in the first two steps compared with the expected depositional biostratigraphic age (112.9 to 96.9 Ma; Araripe & Feijó, 1994), due to collect of  $^{38}Ar$  or  $^{40}Ar$  excess. The age falls into a younger step (about 120 Ma) and then presents an increase in the next ages until it reaches a pseudo-plateau of age about  $377.5 \pm 1.9$  Ma.



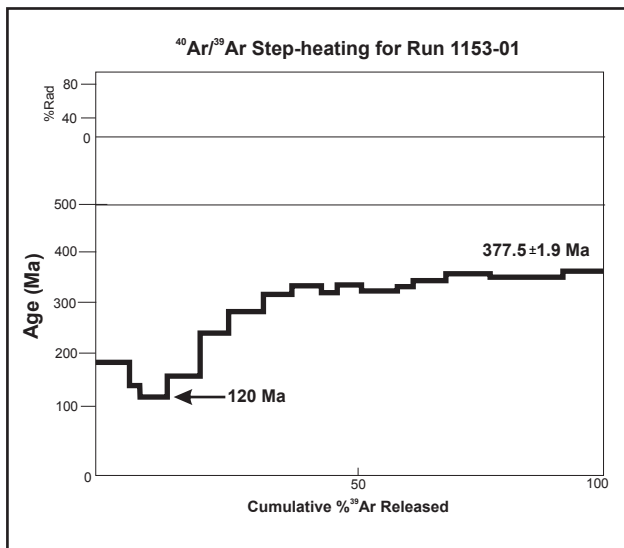


Figure 5.  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  Step-heating spectrum for the grain 1153-01.

The incremental heating spectrum from the second grain (1153-02) does not provide enough detail for an in-depth interpretation and the spectrum is very similar to grain 1153-01. The pseudo-plateau ( $373 \pm 5$  Ma) (Fig. 6) is statistically the same obtained to grain 1153-01. However, a younger age, which could be associated with K-feldspar authigenesis, was not found. This suggests either that was impossible to separate Ar produced from the overgrowths from Ar from the grain through the heating method, or that the volume of overgrowths was too small in this grain.

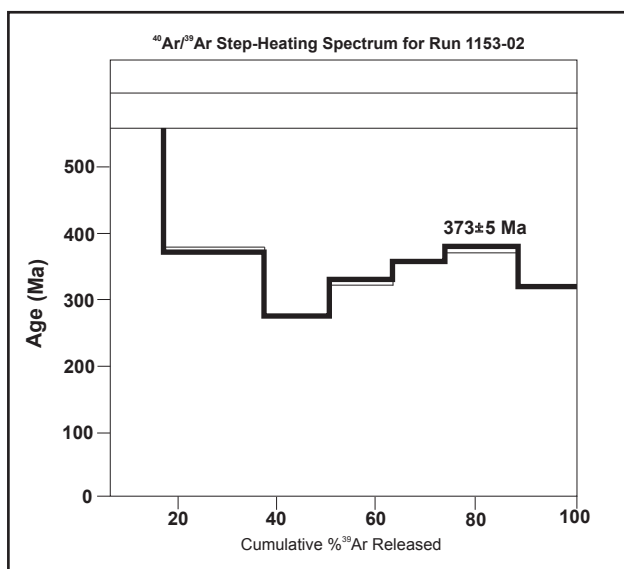


Figure 6.  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  Step-heating spectrum for the grain 1153-02.

The age distribution for the 146 fragments analyzed by the total fusion method is illustrated in the histogram and probability density plots in figure 7. The spread in age ( $428.43 \pm 1.41$  to  $139.17 \pm 0.99$  Ma) represents full range from the age of pure diagenetic feldspar fragments (youngest result), ages of grains containing various proportions of detrital and diagenetic feldspars (intermediate results), and ages of pure detrital cores (oldest results). There are two high probability peaks, one around 170 Ma and the other at about 317 Ma. A noticeable feature in the histogram and probability density plots is the large number of results clustering at *ca.* 300 Ma, which corresponds to the maximum “plateau-like” segment obtained for the incremental heating spectrum for grains 1153-01 and 1153-02.

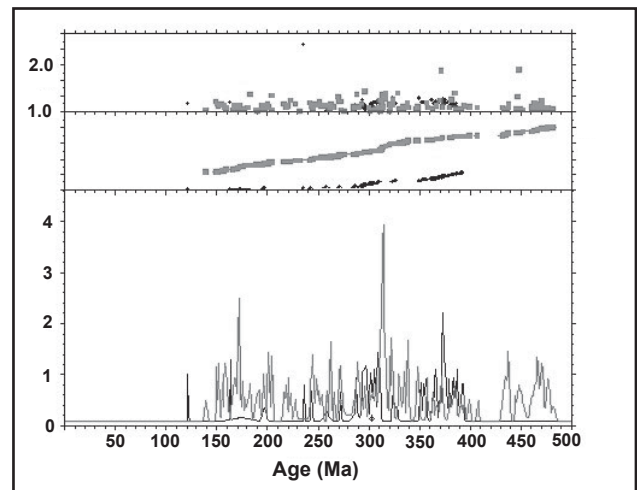


Figure 7. Histogram and Ideogram with all ages of the 146 fragments.

## 5. Discussion

Although early K-feldspar precipitation was already widely reported in several studies (Sibley, 1978; Waugh, 1978; Morad *et al.*, 1989; Girard *et al.*, 1988; Milliken, 1989; Warnock & van de Kamp, 1999; Hagen *et al.*, 2001; Liu *et al.*, 2003; Sandler *et al.*, 2004; Baron *et al.*, 2008), an extremely early and near-surface diagenesis, such as that observed in the Açú Formation sandstones, is rare. No radiometric dating of syngenetic or early diagenetic K-feldspar formed at near-surface temperatures ( $< 50^\circ\text{C}$ ) has yet been published (Sandler *et al.*, 2004). However, the authigenic K-feldspar overgrowths of

the Açú Formation were precipitated soon after deposition and prior to compaction, under direct influence of surface conditions with a temperature around 30<sup>o</sup> C (Maraschin *et al.*, 2004), and before any other diagenetic phase, such as carbonates, mechanically infiltrated clay coatings and iron oxides. The difficulty in separating the detrital grains apart from the overgrowths in the Açú Formation sandstones is probably related to the physical continuity between both phases provided by the lack of intercalated mineral or fluid inclusions, which supposes an early K-feldspar authigenesis. All required ions to form authigenic K-feldspar are supposedly derived from the dissolution of detrital feldspars and micas. This process occurred probably both along the basin margin, coarse-grained, alluvial deposits and in the granitic-gneissic basement rocks overlain by the Açú Formation (Maraschin *et al.*, 2004).

The <sup>40</sup>Ar-<sup>39</sup>Ar results indicate large and consistent differences in ages of detrital grains and authigenic overgrowths. The results indicate a *pseudo-plateau* age for detrital feldspars of 377.5 ± 1.9 Ma, suggesting that some of the detrital feldspars do preserve the age of the source rocks (Borborema granites and gneisses), although older ages between 584 and 495.8 ± 2.5 Ma were obtained from Borborema rocks (Corsini *et al.*, 1998; Neves *et al.*, 2000; Araújo *et al.*, 2005). However, according with Moraes Neto *et al.* (2009), a post-Albian regional uplift affecting the basement source areas, which are consistent with the geologic record in the Araripe Basin, western Borborema Province, indicate that some Ar was lost, consequently with youngest ages in our samples.

Total fusion of the overgrowths samples yielded apparent ages between 139.17 and 120 Ma. Assuming a weak contamination of the overgrowths ages by the detrital cores, the younger age matches with the depositional age available for the Açú Formation (Albo-Cenomanian - from 112.9 to 96.9 Ma; Araripe & Feijó, 1994), estimated from its palynological content. As the laser probe has a large diameter (10-20 µm) relative to the overgrowth size (0.09-0.2 mm width and 0.8 mm length), it is impossible to avoid some contamination by radiogenic Ar from the underlying detrital feldspar. Similar examples described elsewhere (Halliday & Mitchell, 1976; Harrison & McDougall, 1982; Zeitler & Fitzgerald, 1986; Walgenwitz *et al.*, 1990; Girard & Onstott, 1991; Warnock and van de

Kamp, 1999; Hagen *et al.*, 2001) showed that laser bundle can cause Ar loss from grains adjacent to the analyzed area. To establish whether the conductive heating from the laser beam throughout the overgrowth/grain boundary is enough to cause significant degassing of the detrital core is still a question to be properly addressed.

In order to solve this problem, Wartho *et al.* (1999) and Hagen *et al.* (2001) suggest the in situ application of ultraviolet (UV) laser, which is strongly adsorbed by pure minerals, such as authigenic K-feldspars. Also the spatial resolution of the ultraviolet laser is superior to 20 µm for <sup>40</sup>Ar-<sup>39</sup>Ar individual analysis.

The analysis of 146 single fragments yielded an age range from 428.43 ± 1.41 to 139.17 ± 0.99 Ma, which agree well with the end-member ages of detrital grains and overgrowths, respectively.

## 6. Conclusions

Diagenesis of the Açú Formation sandstones is characterized by widespread, very early authigenesis of K-feldspar as overgrowths around detrital microcline and orthoclase grains, which precipitation can be assumed as essentially coeval to deposition. As the physical separation of overgrowths from the feldspar grains was not possible, due to the lack of clays, iron oxides or fluid inclusions between these phases, a conventional <sup>40</sup>Ar-<sup>39</sup>Ar dating method was applied.

The older results obtained owing this work (428.43 ± 1.41 to 373 ± 5 Ma) are compatible with ages for crystals extracted from Borborema granites and gneisses, the source for the main sediments of the Potiguar Basin. Age uniformity is supposed to be related to an uplift event that took place in the region causing Ar loss and age homogenization. This event has also been recorded through dating of the Potiguar Basin basement rocks, the source area of the Açú Formation sandstones.

The minimum age of 120 Ma obtained for the overgrowths is close to that proposed for the deposition of the Açú Formation by Araripe & Feijó (1994), based on palynological data (Albian-Cenomanian; 112.9 to 96.9 Ma). The slightly older age found for some overgrowths suggests a limited Ar contamination from the detrital cores, affected by the relatively large laser beam diameter, what could be overcome by the use of an ultraviolet laser (UV). Other <sup>40</sup>Ar-<sup>39</sup>Ar analyses are suggested for the

Açu Formation sandstones sampled under well-defined stratigraphic control and in different areas of the basin.

The method hereby presented shows great potential for constraining the absolute depositional age of sandstones lacking biostratigraphic resolution but containing early K-feldspar overgrowths.

**Acknowledgements** - The first author is indebted with Brazil National Council for Scientific and Technological Research (CNPq) for the concession of grant 140693-2004-01.

## References

- Ali, A.D. & Turner, P. 1982. Authigenic K-feldspar in the Bromsgrove Sandstones Formation (Triassic) of central England. *Journal of Sedimentary Petrology*, 52: 187-197.
- Almeida, F.F.M., Hasui, Y., Brito-Neves, B.B. & Fuck, R.A. 1981. Brazilian structural provinces: an introduction. *Earth Science Reviews*, 17: 1-29.
- ANP. Agência Nacional do Petróleo. 2005. Available in: <http://www.anp.gov.br/brazil.rounds.gov.br>. Accessed in: março de 2006.
- Araripe, P.T. & Feijó, F.J. 1994. Bacia Potiguar. *Boletim de Geociências da Petrobras*, 8: 27-141.
- Araújo, M.N.C., Vasconcelos, P.M., Silva, F.C.A., de Sá, E.F.J. & Sá, J.M. 2005.  $^{40}\text{Ar}/^{39}\text{Ar}$  geochronology of gold mineralization in Brasiliano strike-slip shear zones in the Borborema province, NE Brazil. *Journal of South American Earth Sciences*, 19: 445-460.
- Baron, M., Parnell, J., Mark, D., Carr, A., Przyjalowski, M. & Feely, M. 2008. Evolution of hydrocarbon migration style in a fractured reservoir deduced from fluid inclusion data, Clair Field, west of Shetland, UK. *Marine and Petroleum Geology*, 25: 153-172.
- Baskin, Y. 1956. A study of authigenic feldspar. *The Journal of Geology*, 64: 132-155.
- Bertani, R.T., Costa, I.G. & Matos, R.M.D. 1990. Evolução tectono-sedimentar, estilo estrutural e habitat do petróleo na Bacia Potiguar. In: Gabaglia, G.P. & Milani, E.J. (Eds.). *Origem e Evolução de Bacias Sedimentares*. Rio de Janeiro, Petrobras, p. 291-310.
- Corsini, M., de Figueiredo, L.L., Caby, R., Féraud, G., Ruffet, G. & Vauchez, A. 1998. Thermal history of the Pan-African/Brasiliano Borborema Province of northeast Brazil deduced from  $^{40}\text{Ar}/^{39}\text{Ar}$  analysis. *Tectonophysics*, 285: 103-117.
- Cremonini, O.A. 1995. A reativação tectônica da Bacia Potiguar no Cretáceo Superior. In: SIMPÓSIO NACIONAL DE ESTUDOS TECTÔNICOS, 5, 1995, Gramado, *Anais... Gramado*, v. 1, p. 277-279.
- De Ros, L.F., Sgarbi, G.N.C. & Morad, S. 1994. Multiple authigenesis of K-feldspar in sandstones: evidence from the Cretaceous Areado Formation, São Francisco Basin, central Brazil. *Journal of Sedimentary Research*, 64: 778-787.
- Falkeinhain, F.U.H., Araújo, M.B. & Souza, S.M. 1977. *Relatório geológico de progresso da Bacia Potiguar*. PETROBRAS, 29p. (Relatório Interno).
- Folk, R.L. 1968. *Petrology of Sedimentary Rocks*. Austin, Texas, Springer, 320p.
- Françolin, J.B. & Sztatmari, P. 1987. Mecanismos de rifteamento da porção oriental da margem norte brasileira. *Revista Brasileira de Geociências*, 17: 196-207.
- Girard, J.P., Aronson, J.L. & Savin, S.M. 1988. Separation, K/Ar dating, and  $^{18}\text{O}/^{16}\text{O}$  ratio measurements of diagenetic K-feldspar overgrowths: an example from the Lower Cretaceous arkoses of the Angola margin. *Geochimica et Cosmochimica Acta*, 52: 2207-2214.
- Girard, J.P. & Onstott, T.C. 1991. Application of  $^{40}\text{Ar}/^{39}\text{Ar}$  laser-probe and step-heating techniques to the dating of diagenetic K-feldspar overgrowths. *Geochimica et Cosmochimica Acta*, 55: 3777-3793.
- Hagen, E., Kelley, S.P., Dypvik, H., Nilsen, O. & Kjolhamar, B. 2001. Direct dating of authigenic K-feldspar overgrowths from the Kilombero Rift of Tanzania. *Journal of Geological Society*, 158: 801-807.
- Halliday, A.N. & Mitchel, J.G. 1976. Structural, K-Ar and  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  age studies of adularia K-feldspar from the Lizard Complex, England. *Earth and Planetary Science Letters*, 29(1): 227-237.
- Harrison, T.M. & McDougall, I. 1982. The thermal significance of potassium feldspar K-Ar ages inferred from  $^{40}\text{Ar}/^{39}\text{Ar}$  age spectrum results. *Geochimica et Cosmochimica Acta*, 46(10): 1811-1820.
- Kastner, M. & Siever, R. 1979. Low temperature feldspar in sedimentary rocks. *American Journal of Science*, 279: 435-479.
- Krainer, K. & Spölt, C. 1989. Detrital and authigenic feldspars in Permian and early Triassic sandstones, eastern Alps (Austria). *Sedimentary Geology*, 62: 59-77.
- Kreidler, W.L. & Andery, P.A. 1949. *Mapa geológico da área sedimentar costeira do Estado do Rio Grande do Norte e Ceará*. Rio de Janeiro, 30p.
- Lee, M. & Savin, S.M. 1985. Isolation of diagenetic overgrowth on quartz sand grains for oxygen isotopic analysis. *Geochimica et Cosmochimica Acta*, 49: 497-501.
- Lee, M.R. & Parsons, I. 2003. Microtextures of authigenic Or-rich feldspar in the Upper Jurassic Humber Group,

- UK North Sea. *Sedimentology*, 50: 597-608.
- Liu, J., Hay, R.L., Deino, A. & Kyser, T.K. 2003. Age and origin of authigenic K-feldspar in uppermost Precambrian rocks in the North American Midcontinent. *Geological Society of America Bulletin*, 115(4): 422-433.
- Maraschin, A.J., Mizusaki, A.M. & De Ros, L.F. 2004. Near-surface K-feldspar precipitation in Cretaceous sandstones from the Potiguar Basin, Northeastern Brazil. *The Journal of Geology*, 112(3): 317-334.
- Maraschin, A.J. & Mizusaki, A.M. 2008. Datação de processos diagenéticos em arenitos-reservatório de hidrocarbonetos: uma revisão conceitual. *Revista Pesquisas em Geociências*, 35(1): 27-41.
- Mark, D.F., Parnell, J., Kelley, S.P., Lee, M., Sherlock, C.S. & Carr, A. 2005. Dating of multistage fluid flow in sandstones. *Science*, 309: 2048-2050.
- Mark, D., Parnell, J., Kelley, S.P. & Sherlock, S.C. 2006. Temperature-composition-time (*T-X-t*) data from authigenic K-feldspar: an integrated methodology for dating fluid flow events. *Journal of Geochemical Exploration*, 89: 259-262.
- Mark, D., Parnell, J., Kelley, S.P. & Sherlock, S.C. 2007. Resolution of regional fluid flow related with successive orogenic events on the Laurentian margin. *Geology*, 35(6): 547-550.
- Mark, D.F., Green, P.F., Parnell, J., Kelley, S.P., Lee, M.R. & Sherlock, S.C. 2008. Late Palaeozoic hydrocarbon migration through the Clair field, West of Shetland, UK Atlantic margin. *Geochimica et Cosmochimica Acta*, 72: 2510-2533.
- Matos, R.M.D. 1987. Sistemas de rifts cretáceos do nordeste brasileiro. In: SEMINÁRIO DE TECTÔNICA DA PETROBRAS (TECTOS I), 1987, Rio de Janeiro, Atas... Rio de Janeiro, p.126-159.
- Milliken, K.L. 1989. Petrography and composition of authigenic feldspars, Oligocene Frio Formation, South Texas. *Journal of Sedimentary Petrology*, 59: 361-374.
- Morad, S., Marfil, R. & la Pena, J.A. 1989. Diagenetic K-feldspar pseudomorphs in the Triassic Buntsandstein sandstones of the Iberian Range, Spain. *Sedimentology*, 36: 635-650.
- Moraes Neto, J.M., Hegarty, K.A., Karner, G.D. & Alkmim, F.F. 2009. Timing and mechanisms for the generation and modification of the anomalous topography of the Borborema Province, northeastern Brazil. *Marine and Petroleum Geology*, 26(7): 1070-1086.
- Neves, S.P., Vauchez, A. & Feraud, G. 2000. Tectono-thermal evolution, magma emplacement, and shear zone development in the Caruaru area (Borborema province, NE Brazil). *Precambrian Research*, 99: 1-32.
- Ruppert, L.F. 1987. Applications of cathodoluminescence of quartz and feldspar to sedimentary petrology. *Scanning Microscopy*, 1: 63-72.
- Saigal, G.C., Morad, S., Bkørlykke, K., Egeberg, P.K. & Aagaard, P. 1988. Diagenetic albitization of detrital K-feldspar in Jurassic, Lower Cretaceous, and Tertiary clastic reservoir rocks from offshore Norway. I. Textures and Origin. *Journal of Sedimentary Petrology*, 58: 1003-1013.
- Sampaio, A.V. & Schaller, H. 1968. Introdução à estratigrafia cretácea da Bacia Potiguar. *Boletim Técnico da Petrobras*, 11: 19-44.
- Sandler, A., Harvalan, Y. & Steinnitz, G. 2004. Early formation of K-feldspar in shallow-marine sediments at near-surface temperatures (southern Israel): evidence from K-Ar dating. *Sedimentology*, 51: 323-338.
- Santos, E.J. & Brito-Neves, B.B. 1984. Província Borborema: In: Almeida, F.F.M. & Hasuy, Y. (Eds.). *O Pré-Cambriano do Brasil*. São Paulo, Edgard Blücher, p. 123-186.
- Sherlock, S.C., Lucks, T., Kelley, S.P. & Barnicoat, A. 2005. A high resolution record of multiple diagenetic events: ultraviolet laser microprobe Ar/Ar analysis of zoned K-feldspar overgrowths. *Earth and Planetary Science Letters*, 238: 329-341.
- Schobbenhaus, C., Campos, D.A., Derze, G.R. & Asmus, H.E. 1984. Geologia do Brasil: texto explicativo do mapa geológico do Brasil e da área de ocorrência incluindo depósitos minerais, escala 1:2.500.000. Brasília, Ministério de Minas e Energia / Departamento Nacional de Produção Mineral, 501p.
- Sial, A.N. 1986. Granite-types in northeast Brazil: current knowledge. *Revista Brasileira de Geociências*, 16: 54-72.
- Sibley, D.F. 1978. K-feldspar cement in the Jacobsville sandstone. *Journal of Sedimentary Petrology*, 48: 983-986.
- Smith, J.V. & Stenstrom, R.C. 1965. Electron-excited luminescence as a petrologic tool. *Journal of Geology*, 73: 627-653.
- Souto Filho, J.D., Correa, A.C.F., Santos Neto, E.V. & Trindade, L.A.F. 2000. Alagamar-Açu petroleum system, onshore Potiguar Basin, Brazil: A numerical approach for secondary migration. In: Mello, M.R. & Katz, B.J. (eds.). *Petroleum Systems of South Atlantic Margins*. AAPG Memoir 73, p.151-158.
- Souza, S.M. 1982. Atualização da litoestratigrafia da Bacia Potiguar. In: CONGRESSO BRASILEIRO DE GEOLOGIA, 32, 1982, Salvador. *Anais...* Salvador, SBG v.31(5), p.2392-2406.
- Stablein III, N.K. & Dapples, E.C. 1977. Feldspars of the Tunnel City Group (Cambrian), western Wisconsin.



- Journal of Sedimentary Petrology*, 47: 1512-1538.
- Vasconcelos, E.P., Lima Neto, F.F. & Ross, S. 1990. Unidades de correlação da Formação Açú, Bacia Potiguar. In: CONGRESSO BRASILEIRO DE GEOLOGIA, 36, 1990, Natal. *Anais...* Natal, SBG, v. 36(1), p. 227-240.
- Vasconcelos, P.M., Onoe, A.T., Kawashita, K., Soares, A.J. & Teixeira, W. 2002.  $^{40}\text{Ar}/^{39}\text{Ar}$  geochronology at the Instituto de Geociências, USP: instrumentation, analytical procedures, and calibration. *Anais da Academia Brasileira de Ciências*, 74(2): 297-342.
- Walgenwitz, P., Pagel, M., Meyer, A., Maluski, H. & Moine, P. 1990. Thermo-chronological approach to reservoir diagenesis in the offshore Angola Basin: a fluid inclusion, Ar-Ar and K-Ar investigation. *American Association Petroleum Bulletin*, 74(5): 547-563.
- Warnock, A.C. & van de Kamp, P.C. 1999. Hump-shaped  $^{40}\text{Ar}$ - $^{39}\text{Ar}$  age spectra in K-feldspar and evidence for Cretaceous authigenesis in the Fountain Formation near Eldorado Springs, Colorado. *Earth and Planetary Science Letters*, 174: 99-111.
- Wartho, J.-A., Kelley, S.P., Brooker, R.A., Carroll, M.R., Villa, I.M. & Lee, M.R. 1999. Direct measurement of Ar diffusion profiles in a gem-quality Madagascar K-feldspar using the ultra-violet laser ablation microprobe (UVLAMP). *Earth and Planetary Science Letters*, 170: 141-153.
- Waugh, B. 1978. Authigenic feldspar in British Permian-Triassic sandstones. *Journal of the Geological Society*, 135: 51-56.
- Zeitler, P.K. & Fitzgerald, J.D. 1986. Saddle-shaped  $^{40}\text{Ar}/^{39}\text{Ar}$  age spectra from young, microstructurally complex potassium feldspars. *Geochimica et Cosmochimica Acta*, 50: 1185-1199.