Chronostratigraphic framework and provenance of the Ossa-Morena Zone Carboniferous basins (SW Iberia)

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Abstract. Carboniferous siliciclastic and silicic magmatic rocks from the Santa Susana-São Cristovão region contain valuable information regarding the timing of synorogenic processes in SW Iberia. In this region of the Ossa-Morena Zone (OMZ), Late Carboniferous terrigenous strata (i.e. the Santa Susana Formation) unconformably overlie Early Carboniferous marine siliciclastic deposits alternating with volcanic rocks (i.e. the Toca da Moura volcano-sedimentary complex). Lying below this intra-Carboniferous unconformity, the Toca da Moura volcano-sedimentary complex is intruded and overlain by the Baleizão porphyry. Original SHRIMP and LA-ICP-MS U-Pb zircon are presented in this paper, providing chronostratigraphic and provenance constraints, since available geochronological information is scarce and only biostratigraphic ages are currently available for the Santa Susana-São Cristovão region. Our findings and the currently-available detrital zircon ages from Paleozoic terranes of SW Iberia (Pulo do Lobo Zone- PLZ, South-Portuguese Zone- SPZ, and OMZ), were jointly analyzed using the K-S test and MDS diagrams to investigate provenance. The marine deposition is constrained to the age interval of c. 335-331 Ma (Visean) by new U-Pb data for silicic tufts from the Toca da Moura volcano-sedimentary complex. The Baleizão porphyry, intrusive in the Toca da Moura volcano-sedimentary complex, yielded a crystallization age of c. 317 Ma (Bashkirian), providing the minimum age for the overlying intra-Carboniferous unconformity. A comparison of detrital zircon populations from siliciclastic rocks of the Cabrela and Toca de Moura volcano-sedimentary complexes of the OMZ suggests that they
derived from distinct sources more closely associated with the SPZ and PLZ than the OMZ.

Above the intra-Carboniferous unconformity, the Santa Susana Formation is either the result of
the recycling of distinct sources located in the Laurussian-side (SPZ and PLZ) and Gondwanan-
side (OMZ) of the Rheic suture zone. The best estimate of the crystallization age of a granite
cobble found in a conglomerate from the Santa Susana Formation yielded c. 303 Ma
(Kasimovian-Gzhelian), representing the maximum depositional age for the terrestrial strata.

The intra-Carboniferous unconformity seems to represent a stratigraphic gap of approximately
12-14 Ma, providing evidence of the rapid post-accretion/collision uplift of the Variscan
orogenic belt in SW Iberia (i.e. the OMZ, PLZ and SPZ).

1. Introduction

U-Pb geochronology of detrital zircon from siliciclastic rocks has been extensively used in
stratigraphic correlation studies for estimating the maximum depositional age and investigating
the provenance of sedimentary sequences (Fedo et al., 2001; Dickinson and Gehrels, 2009). The
youngest detrital zircon grains found in siliciclastic rock commonly provide useful information
about depositional age, especially in areas that experienced active volcanism during sediment
accumulation (Gehrels, 2014). The maximum depositional age obtained for siliciclastic rock is
often not necessarily coincident with the biostratigraphic age as defined by key fossil
assemblages (Pereira et al., 2019). Therefore, in order to overcome any doubt about the true age
of deposition, it is desirable that volcanic rocks interstratified with fossiliferous siliciclastic
rocks should be dated (Fedo et al., 2001; Bowring et al., 2006). Furthermore, the application of
zircon U-Pb geochronology to volcano-sedimentary and sedimentary sequences that are
separated by unconformities, by means of the comparative analysis of their age populations,
may be useful for estimating time intervals and revealing changes in provenance. Volcanic
rocks that lie beneath or overlie sedimentary sequences and unconformities can provide
maximum and minimum ages, respectively. When detrital zircon geochronology is linked to the
geochronology of crosscutting younger igneous rocks, then both a maximum and minimum age
bracket for deposition can be determined (Fedo et al., 2001).

The Variscan orogen that extends from central Europe to Iberia was reworked through discrete
Carboniferous sedimentary cycles during the Laurussia-Gondwana convergence, giving rise to
the formation of marine and terrestrial basins. In SW Iberia, stratigraphic correlation has been
proposed for the Carboniferous synorogenic strata found in the three main tectonostratigraphic
divisions of the Variscan Orogen: the Ossa-Morena (OMZ), Pulo do Lobo (PLZ) and South
Portuguese (SPZ) zones (Quesada and Oliveira, 2019, and references therein).

The Carboniferous siliciclastic strata in the Santa Susana-São Cristovão region (OMZ) includes
fossils indicating Carboniferous to Kasimovian biostratigraphic ages (Teixeira, 1938-1940, 1941;
Lemos de Sousa and Wagner, 1983; Wagner and Lemos de Sousa, 1983; Pereira et al., 2006;
Machado et al., 2012; Lopes et al., 2014). In the Santa Susana-São Cristovão region, Late Carboniferous siliciclastic strata of the Santa Susana Formation unconformably overlie: i) the poorly-dated Baleizão volcanic-subvolcanic suite, and ii) the Early Carboniferous Toca da Moura volcano-sedimentary complex, which includes volcanic rocks that have never been dated. This intra-Carboniferous unconformity was generated as consequence of regional uplift and falling sea level, leading to a change in depositional environment from Early Carboniferous marine to Late Carboniferous terrestrial (Gonçalves and Carvalhosa, 1984; Oliveira et al., 1991; Machado et al., 2012). The provenance of the above-mentioned Carboniferous strata has been discussed based on petrographic, paleontological and detrital zircon geochronology evidence (Pereira et al., 2006; Machado et al., 2012; Lopes et al., 2014; Dinis et al., 2018).

In this paper, SHRIMP and LA-ICP-MS U-Pb analyses were performed on zircon grains from silicic volcanic, subvolcanic, and siliciclastic rocks sampled in the Santa Susana-São Cristovão region (OMZ, SW Iberia). The aim of this geochronology study is to establish the chronostratigraphic framework of the Carboniferous strata in the Santa Susana-São Cristovão region and to discuss their provenance using a statistical approach (Kolmogorov-Smirnov test and Mutiscaling diagrams). Thus we pay tribute to J.R. Martínez-Catalán, who devoted part of his career to investigating the Carboniferous synorogenic basins of NW Iberia.

2. Geological setting

In SW Iberia, the tectonic limit between the OMZ (Gondwanan-side) and the PLZ and SPZ (Laurussian-side) has been regarded as constituting the tectonically reworked suture zone of the Rheic Ocean (Andrade, 1983; Quesada et al., 1994; Simancas et al., 2005; Díaz-Apiroz et al., 2006; Ribeiro et al., 2007; Pereira et al., 2017a) (Fig. 1). This Paleozoic suture zone has been defined along the Beja-Acebuches ophiolitic complex (Fonseca et al., 1999, and references therein). The Beja-Acebuches ophiolitic complex is separated from the Beja Igneous Complex (Jesus et al., 2007, 2016) by a strike-slip fault. Metabasalts and meta-gabbros (i.e. the Mombeja unit of Andrade, 1983) from the Beja-Acebuches ophiolitic complex have been dated at c. 340-332 Ma (U-Pb zircon; Azor et al., 2008), while in the Beja Igneous Complex gabbro and granitic rocks are relatively older, yielding crystallization ages of c. 353-342 Ma (U-Pb zircon; Jesus et al., 2007; Pin et al., 2008). Trace element and isotopic signatures of Beja Igneous Complex plutonic rocks indicate crustal contamination of parental magmas deriving from a depleted asthenospheric mantle reservoir (Santos et al., 1990; Pin et al., 2008; Jesus et al., 2016). The plutonic rocks of the Beja Igneous Complex show well-defined intrusive contacts with previously deformed and metamorphosed sedimentary and igneous rocks of the OMZ basement (Rosas et al., 2008; Pin et al., 2008). The Beja Igneous Complex also includes the São Cristovão-Alcâçovas subvolcanic complex (Gonçalves and Carvalhosa, 1984), composed of silicic sub-volcanic and volcanic rocks (i.e. the Baleizão unit of Andrade, 1983), granophyres
and porphyries dated at c. 324 Ma (K-Ar on biotite; Priem et al., 1986), associated with diabases. The major and trace element geochemistry of the Baleizão porphyries indicates a calc-alkaline rhyolitic, rhyodacitic and andesitic composition typical of magmas produced at convergent plate boundaries (Santos et al., 1987; Caldeira et al., 2007; Ferreira et al., 2014). The Baleizão porphyries occur as dykes and sills (Andrade, 1927) (Figs. 3a, b), overlying (Gonçalves and Carvalhosa, 1984) the Early Carboniferous siliciclastic and volcanic rocks of the Toca da Moura volcano-sedimentary complex (Santos et al., 1987, and references therein) (Fig. 2).

The Toca da Moura volcano-sedimentary complex is mainly composed of pelites (i.e. “Xistinhos”; Teixeira, 1944; Fig. 3a) and greywackes, associated with andesite-to-rhyolite volcanic rocks (lava flow and tuffs; Figs. 3c, d, e), andesitic basalt (Fig. 3f), chert layers (Gonçalves and Carvalhosa, 1984), and a few olistoliths of basalt and limestone. Siliciclastic rocks contain well-preserved in-situ palynomorph assemblages of Tournaisian to Visean age and reworked palynomorphs ranging in age from the Middle Cambrian to the Early Tournaisian (Pereira et al., 2006; Lopes et al., 2014). Based on geochemical information, this volcanism was interpreted by Santos et al. (1987) as deriving from calc-alkaline magma produced in a continental magmatic arc. A stratigraphic correlation was established between the Toca da Moura volcano-sedimentary complex and the Cabrela volcano-sedimentary complex (Pereira et al., 2006) which is located 15 km to the NW, in the Évora Massif (Pereira et al., 2007; 2012a) (Fig. 1b). The presence of variable-scale soft-sediment structures (i.e. slumps, intraclast conglomerates and olistoliths) in both complexes indicates gravity-induced instability during marine sedimentation. Detrital zircon ages of a siliciclastic rock from the Cabrela volcano-sedimentary complex are mainly Middle-Late Devonian (82%) and Early Carboniferous (14%), also including a few older grains (sample OM-200 from Pereira et al., 2012a).

The Santa Susana Formation (i.e. Santa Susana basin, Domingos et al., 1983; Quesada et al., 1990, Oliveira et al. 1991) siliciclastic rocks that outcrop along a NNW-SSE-trending narrow discontinuous band which is 0.1-5 km wide and 12 km long unconformably overlie the Baleizão Porphyry and the Toca da Moura volcano-sedimentary complex (Fig. 2), forming the geological contact between these stratigraphic units often defined by faults (Gonçalves and Carvalhosa, 1984). The Santa Susana Formation is divided into two members (Machado et al., 2012, and references therein): i) the lower member is mainly composed of coarse-grained sandstone and conglomerate beds (Figs. 4a, b, c, d); these conglomerates include pebbles and cobbles of silicic porphyry, rhyolite, andesite, basalt, granite, felsic tuff, pelite, sandstone, greywacke, quartzite, phyllite, chert, and quartz (Figs. 4e, f); ii) the upper member represents a repetitive sequence of alternating beds of pelite and sandstone interbedded with coal seams, and few beds of conglomerate (Fig. 2). These terrestrial deposits were most probably deposited in an alluvial/fluvial-to-fluvial/lacustrine (floodplain lakes and/or abandoned channels with abundant
vegetation) system. The plant fossils identified in the siliciclastic rocks of the Santa Susana Formation indicate a Moscovian-Kasimovian biostratigraphic age (Wagner and Lemos de Sousa, 1983). Pelitic beds from the Upper member include palynomorph assemblages assigned with Kasimovian age (Machado et al., 2012). Palynomorphs ranging in age from the middle Cambrian to the early Moscovian were also found in siliciclastic rocks of the Santa Susana Formation sampled from a borehole at a depth of around 400 m (Lopes et al., 2014). Detrital zircon ages from upper member sandstones (Dinis et al., 2018) are mainly distributed over Devonian-Carboniferous (41-51%), Paleoproterozoic (23-30%) and Ediacaran-Cryogenian (16-23%) groups, and also a few Stenian-Tonian and Archean grains.

3. Rational and analytical methods

In this study, SHRIMP U-Pb analyses were performed for the first time on magmatic zircon from two samples of tuff from the Toca da Moura volcano-sedimentary complex (TM-1 and SCV-2; Figs. 3c, d), one from the Baleizão silicic porphyry (SCV-30; Fig. 3b), and a cobble of granite (SCV-7; Fig. 4e) found in a conglomerate from the lower member of the Santa Susana Formation. Estimations of the crystallization age of samples SCV-2 and TM-1 (syndepositional volcanism), and sample SCV-30 (post-depositional) were used to validate the Tournaisian-Visean biostratigraphic age previously attributed to the Toca da Moura volcano-sedimentary complex based on palynological assemblages (Pereira et al., 2006; Lopes et al., 2014). The presence of granite cobbles and pebbles in conglomerate layers from the lower Santa Susana Formation indicates denudation and recycling of a crystalline basement involving granite whose age is unknown. The dating of the granite cobble (sample SCV-7) is useful for discussing provenance and estimating the maximum depositional age of the Santa Susana conglomerate. In addition, LA-ICP-MS U-Pb analyses were performed on detrital zircon grains from two samples of sandstone from the upper and lower members of the Santa Susana Formation (samples SS-1 and SS-2, respectively; Fig. 5g, h), and a sample of pelite from the Toca da Moura volcano-sedimentary complex (sample TM-3; Fig. 5e). This new U-Pb data is useful for discussing provenance and determining the maximum depositional ages of the two sedimentary sequences separated by an intra-Carboniferous unconformity. Sample locations in the Santa Susana-São Cristovão region are indicated in Figure 2. Finally, detrital zircon grains of siliciclastic rock from the Cabrela volcanic-sedimentary complex (sample CBR-11; Fig. 5f; equivalent to sample OM-200 of Pereira et al. 2012a) were analyzed to test for the existence of pre-Devonian ages. The new U-Pb results obtained in the present study are compared with previously-reported age spectra for pre-Kasimovian siliciclastic rocks from the OMZ, PLZ and SPZ siliciclastic sequences of SW Iberia, using statistical tools.

Zircon grains for U-Pb geochronology were selected using traditional techniques: density separation using a willfley table (Universidad Complutense de Madrid, Spain) and also using...
granulometric separation using sieves with a mesh size of less than 500 microns, density (panning) separation procedures, and mineral identification using a binocular lens and preparation of epoxy resin mounts with zircon grains (Universidade de Évora, Portugal). U-Pb measurements were obtained at IBERSIMS (Universidad de Granada, Spain) using SHRIMP, and also at the Senckenberg Naturhistorische Sammlungen Dresden (Museum für Mineralogie und Geologie, Germany) using a LA-ICP-MS. U-Pb measurements using SHRIMP and LA-ICP-MS followed the procedures previously described by Dias da Silva et al. (2018) and Pereira et al. (2012a), respectively. U-Pb results are listed in Tables S1 and S2 (Supplementary Material). Concordia curves and weighted-average means were obtained using Isoplot 4 (Ludwig, 2003) (Figs. 6 and 7). Kernel density estimation (KDE) diagrams were produced with 90-110 % concordant $^{206}\text{Pb}/^{238}\text{U}$ ages for grains younger than 1.0 Ga, and $^{207}\text{Pb}/^{206}\text{Pb}$ ages for older grains (for further details, see Frei and Gerdes, 2009) using IsoplotR (Vermeesch, 2018) (Figs. 8a, b). Cathodoluminescence-imaging was performed at TU Bergakademie Freiberg (Germany) and at IBERSIMS.

The K-S test and the MDS technique were used in conjunction to compare populations of detrital zircon U-Pb ages obtained from the Carboniferous siliciclastic rocks of the Santa Susana-São Cristovão region using a method designed for a recent study of the provenance of Triassic sandstones (Gama et al., in press, and references therein). The K-S test is a non-parametric statistical tool that has been successfully used for the comparison of two populations of detrital zircon U-Pb ages by evaluating whether they are significantly different, i.e. indicating whether zircon age populations correlate with a similar source or not, regardless of whether they are of different sizes, while including at least 20 measurements (DeGraaff-Surpless et al., 2003). The probability of the observed maximum vertical difference between the cumulative probability curves (D-value; Fig. 7c) being unrelated to age differences between the two detrital zircon populations is given by a P-value corresponding to a confidence interval of 95% (Barbeau Jr. et al.; 2009; Guynn and Gehrels, 2010) (Fig. 9a). High P-values and low D-values indicate that the observed difference between the two detrital zircon populations may be explained by the existence of common sources (Gama et al., in press, and references therein). K-S analyses were carried out using an Excel spreadsheet published on the University of Arizona Geochronological Center website at https://sites.google.com/a/laserchron.org/laserchron/. The MDS technique provides a means for the comparison of samples based on quantified pairwise comparisons of their detrital zircon ages, and is extremely useful for visualising the degree of similarity between samples in two dimensions, i.e. greater distances between samples represent a greater degree of dissimilarity between points on MDS diagrams (Vermeesch, 2013; Spencer and Kirkland, 2015; Wissink et al., 2018) (Fig. 9b). MDS diagrams were produced using IsoplotR (Vermeesch, 2018).
4. U-Pb geochronology: Results

4.1. Volcanic silicic rocks of the Toca da Moura volcano-sedimentary complex

Sample SCV-2 is a fine-grained banded rhyolitic tuff consisting of variable size and shape quartz and K-feldspar phenocryst and lithoclasts (less than 1 mm in diameter) dispersed in ash matrix (Fig. 5a). Zircon grains appear as stubby-to-elongated euhedral prisms (50-150 μm in diameter), mostly showing oscillatory concentric zoning growing on distinct cores or as simple crystals. There are some dark inclusions, unzoned patches and transgressive variably luminescence embayments. A total of 44 U-Th-Pb SHRIMP analyses of 44 grains yielded U content ranging from 262 to 628 ppm, and Th/U ratios ranging from 0.17 to 0.95 (mean = 0.42). A group of 23 grains with \(^{206}\text{Pb}/^{238}\text{U}\) ages (discordance ≤ 5%) yielded a weighted mean \(^{208}\text{Pb}/^{238}\text{U}\) age of 331 ± 4 Ma (MSWD = 1.2; Fig. 6a), which probably represents the crystallization age of tuff.

Sample TM-1 is a fine-grained banded rhyolitic tuff consisting quartz, K-feldspar and biotite phenocrysts, flattened dark-brown pumice (i.e. fiamme) and lithoclasts (less than 1 mm in diameter) enclosed in ash matrix (Fig. 5b). The zircon population is characterized by stubby euhedral-to-sub-euhedral small (30-100 μm in diameter) grains. Magmatic grains are either simple with concentric zoning or composite showing variably luminescence cores with concentric zoning, unzoned, or banded zoned. These cores are surrounded by overgrowths with concentric zoning and are occasionally diffuse or unzoned. A total of 120 U-Th-Pb LA-ICP-MS analyses yielded U content ranging from 87 to 4136 ppm, and Th/U ratios ranging from 0.04 to 2.29 (mean = 0.53). 28 \(^{206}\text{Pb}/^{238}\text{U}\) ages (90-110% of concordance) yield a weighted mean \(^{208}\text{Pb}/^{238}\text{U}\) age of 341 ± 10 Ma with a very poor fit (MSWD = 6.9; Fig. 6b), as indicated by the scattering of ages along the Concordia curve. A coherent group of 21 grains with \(^{206}\text{Pb}/^{238}\text{U}\) ages yielded a weighted mean \(^{208}\text{Pb}/^{238}\text{U}\) age of 335 ± 6 Ma (MSWD = 1.5; Fig. 6b), providing the best age estimate for the volcanic rock (Fig. 6b). The youngest zircon grain (c. 302 Ma) probably experienced Pb loss. The six oldest zircon grains present Paleoproterozoic (c. 2 Ga), Neoproterozoic (c. 715 Ma) and Devonian (c. 395-378 Ma) ages, suggesting inheritance.

4.2. Baleizão porphyry

Sample SCV-30 is a porphyritic rhyodacite-rhyolite consisting of quartz, plagioclase, K-feldspar, biotite and amphibole phenocryst (less than 3 mm in diameter) embedded in a fine-grained silicic matrix (Fig. 5c). The zircon population contains grains (30-120 μm in diameter) from subrounded subhedral to prismatic euhedral. Prisms are equant to moderately elongate showing simple internal structure characterized by concentric and sector zoning to unzoned. A concentric zoned or unzoned rim surrounds unzoned cores of few composite grains. A total of 20 U-Th-Pb SHRIMP analyses for sample SCV-30 yielded U content ranging from 267 to 581 ppm, and Th/U ratios ranging from 0.34 to 0.52 (mean = 0.41). 15 analyses were obtained for
zircon with discordance ≤ 5%, distributed along the concordia curve from ca. 355 to 312 Ma, and yielded a weighted mean $^{206}\text{Pb}^{238}\text{Th}$ age of 332 ± 9 Ma (mean square of weighted deviates, MSWD = 4.3; Fig. 7b). Some of the spread observed could be due to the presence of inheritance. Five grains in the age range ca. 334-312 Ma yielded a weighted mean $^{206}\text{Pb}^{238}\text{U}$ age of 317 ± 12 Ma (MSWD = 2.1; Fig. 7a), which is regarded as the best estimate for the crystallization age of subvolcanic silicic rock. The remaining oldest 10 grains yielded $^{206}\text{Pb}^{238}\text{U}$ ages of c. 355-337 Ma which probably indicates inheritance.

4.3. Cobble of granite found in a conglomerate from the Santa Susana Formation

Sample SCV-7 is a cobble (20 cm in diameter) of pinkish medium-grained granite consisting of quartz, alkali feldspar and biotite (Fig. 5d). Most zircons are stubby and elongated subeuhedral to euhedral prisms (80 to 150 μm in diameter). Morphologically zircon grains are mostly simple showing concentric zoning, sector zoning to unzoned, and few are composite with irregular and unzoned small cores surrounded by a rim with concentric zoning. 40 U-Th-Pb SHRIMP analyses were performed on sample SCV-7 with U content ranging from 348 to 3177 ppm, and Th/U ratios ranging from 0.3 to 1.25 (mean = 0.5). Of this total of analyses 24 U-Pb ages with discordance ≤ 5%, scattered along the concordia curve from ca. 349 to 294 Ma, yielded a weighted mean $^{206}\text{Pb}^{238}\text{U}$ age of 327 ± 7 Ma (MSWD = 4; Fig. 7b). A group of six zircon grains in the age range of c. 309-294 Ma yielded a weighted mean $^{206}\text{Pb}^{238}\text{U}$ age of 303 ± 6 Ma (MSWD = 0.98; Fig. 7b), which is taken as the probable crystallization age of the granite. The remaining 19 zircon grains yielded $^{206}\text{Pb}^{238}\text{U}$ ages of c. 349-326 Ma, suggesting inheritance.

4.4. Siliciclastic rocks from the Toca da Moura and Cabrela volcano-sedimentary complexes

Sample TM-3 is a laminated poorly-sorted siltstone with quartz-rich silt layers, containing feldspar and tourmaline grains, and lithoclasts (Fig. 5e), which are intercalated with darker layers of clay. The zircon population is mostly characterized by stubby to elongated prismatic small grains (less than 100 μm in diameter). It includes simple and composite zircons showing concentric, sector and banded zoning. Of a total of 82 U-Th-Pb LA-ICP-MS analyses, with U content ranging from 19 to 4630 ppm, and Th/U ratios ranging from 0.01 to 4.53 (mean = 0.75), 36 zircon grains yield 90-110% concordance. The Paleozoic population of detrital zircon (36%) includes Early Carboniferous (9%, c. 353, 349 and 340 Ma), Ordovician (14%, c. 476-456 Ma), Cambrian (7%, c. 531-500 Ma) and Late Devonian (6%, c. 369 and 362 Ma) grains (Fig. 8a).

The Precambrian population (64%) is predominantly Neoproterozoic (36%; c. 983-587 Ma), but also includes Paleoproterozoic (14%; c. 2.1-1.8 Ga), Mesoproterozoic (8%; c. 1.3-1 Ga) and Archean (6%; c. 2.7-2.5 Ga) grains. The three youngest zircon grains (c. 353-340 Ma) yielded a maximum depositional age of c. 348 Ma (Tournaisian), which is in accordance with the
sedimentary age inferred from biostratigraphic constraints (Late Tournaisian to Middle-Late Viséan; Pereira et al., 2006; Lopes et al., 2014).

Sample CBR-11 is a fine-grained poorly-to-moderate sorted siltstone consisting predominantly of quartz and few feldspar grains and lithoclasts enclosed in silt-clay-sized particles (Fig. 5g). Most of zircon grains are small (less than 100 μm in diameter), euhedral to subeuhedral. They are simple grains (short, stubby to equant prisms) with oscillatory concentric and banded zoning, and only few are composite grains with rounded cores. Of a total of 20 U-Th-Pb LA-ICP-MS analyses, with U content ranging from 54 to 1379 ppm, and Th/U ratios ranging from 0.2 to 1.69 (mean = 0.81), 10 grains yielded 90-110% of concordance. Five grains are Paleozoic (Carboniferous: c. 359, 351 and 346 Ma; Cambrian: c. 514 and 511 Ma) and five are Precambrian (Paleoproterozoic: c. 2.4, 2.1 and 1.8 Ga; Mesoproterozoic: 1 Ga; Neoproterozoic: c. 603 Ma). By combining our new data with those from sample OM-200 (Pereira et al., 2012a), it was found that the detrital zircon population (CB; Fig. 8a) is largely dominated by Paleozoic grains (90%): Late-Middle Devonian (68%), Early Carboniferous (15%), Cambrian (4%) and Early Devonian (2%) grains, being distinct from sample TM-3 described above (Fig. 8a). The youngest zircon population (N = 5; c. 353-346 Ma) yielded a weighted mean age of c. 351 Ma (Tournaisian), suggesting a maximum depositional age which is slightly older than the sedimentary age inferred from biostratigraphic constraints (Late Tournaisian to Middle-Late Viséan; Pereira et al., 2006).

4.5. Siliciclastic rocks from the Santa Susana Formation

Sample SS-2 represents medium-to-coarse grained poorly-sorted sandstone. It is mainly composed of lithoclasts (siltstone, mudstone, quartzite, phyllite, rhyolite, basalt) and quartz grains, but also includes muscovite and feldspar grains (Fig. 5g). The zircon population is mostly characterized by stubby to prismatic, subrounded to subangular, grains (120-300 μm in diameter). Morphologically were found simple and composite grains. Cathodoluminescence imaging shows that most zircon grains have concentric oscillatory zoning, irregular zoning and are banded or unzoned. A total of 153 U-Th-Pb LA-ICP-MS analyses were performed on detrital zircon grains. They show U content ranging from 15 to 6158 ppm, and Th/U ratios ranging from 0.02 to 3.57 (mean = 0.66). A population with 51 grains yielding U-Pb ages with 90-110% concordance (Fig. 8b) is dominated by Precambrian ages (64%): Neoproterozoic (37%; c. 801-551 Ma), Paleoproterozoic (25%; c. 2.4-1.6 Ga) and Neorchean (2%, c. 2.5 Ga).

The Paleozoic grains (36%) are Carboniferous (20%; c. 359-303 Ma), Late Devonian (14%; c. 378-362 Ma), and Early Ordovician (2%; c. 447 Ma). The youngest grain (c. 303 Ma; Kasimovian-Gzhelian) is slightly younger than the sedimentary age inferred from biostratigraphic constraints (Middle Moscovian to Kasimovian; Lemos de Sousa and Wagner, 1983; Machado et al., 2012; Lopes et al., 2014).
Sample SS-1 represents a very-coarse grained sandstone consisting of rounded-to-subangular mono- and polycrystalline quartz, feldspar and muscovite grains, and a wide variety of lithoclasts (chert, phyllite, rhyolite, siltstone and sandstone; Fig. 5h). Zircon grains are rounded to subangular, stubby and elongated prisms (less than 280 μm in diameter). The zircon population includes simple grains with oscillatory concentric, banded and sector zoning, and composite grains with cores with distinct internal morphologies surrounded by variable width rims. A total of 150 U-Th-Pb LA-ICP-MS analyses performed on detrital zircon grains yielded U content ranging from 24 to 9819 ppm, and Th/U ratios ranging from 0.05 to 2.89 (mean = 0.72). A group of 71 grains yielding U-Pb ages with 90-110% concordance are dominated by Paleozoic ages (82%), predominantly made up of Carboniferous (49%; c. 358-315 Ma) and Devonian (25%; c. 389-359 Ma), and a few Late Ordovician-Silurian (5%; c. 434, 429 and 425 Ma) and Cambrian (3%; c. 533 and 491 Ma) grains (Fig. 8b). The Precambrian grains (18%) are Neoproterozoic (10%; c. 702-542 Ma), Paleoproterozoic (4%; c. 2.1-1.6 Ga), Mesoproterozoic (3%, c. 1.4 and 1.6 Ga) and Neorchean (1%, c. 2.8 Ga). The youngest zircon population (N = 3; c. 319-315 Ma) yielded a maximum depositional age of c. 316 Ma (Bashkirian-Moscovian), which is slightly older than the sedimentary age inferred from biostratigraphic constraints (Middle Moscovian to Kasimovian; Lemos de Sousa and Wagner, 1983; Machado et al., 2012; Lopes et al., 2014).

5. K-S test and MDS analysis: results

The K-S test performed on the Santa Susana sandstones show that the detrital zircon populations of sample SS-2 (lower member) and SS upper member (i.e. includes samples StSz2 and StSz4 from Dinis et al., 2018) are ‘not significantly different’ (all ages- P-value = 0.169; pre-Carboniferous ages- P-value = 0.879) at the 5% confidence level (Fig. 9a). A comparison of samples SS-1 and SS-2 reveals that they are “significantly different” (P-value ≤ 0.01). Unlike sample SS-2, the sample SS-1 detrital zircon population is “significantly different” (P-value < 0.01) from the SS upper population (Fig. 9a), indicating distinct sources. Besides this, sample SS-1 is much closer to that of the SS upper (D-value = 0.323), and more distant from sample SS-2 (D-value = 0.465) as regards the distance between cumulative probability curves (Fig. 8c).

In Figure 9b, the MDS diagram produced with all ages shows sample SS-1 adjacent to Cabrela and Mértola siliciclastic rocks, while sample SS-2 is near the Mira, Santa Iria and Represa detrital zircon populations. In the MDS diagram for pre-Carboniferous ages, sample SS-2 is juxtaposed with sample TM-3, and closest to the Mira, Phyllite-Quartzite and Tercenas formations (Fig. 9c) suggesting likely sources. Nevertheless, the probable contribution to SS-2 samples of sediment derived from the oldest siliciclastic rocks from the PLZ and SPZ (i.e. Pulo do Lobo, Gafo, Ribeira de Limas, Atalaia and Ronquillo formations), and OMZ sources cannot be excluded. Their detrital zircon populations are ‘not enough significantly different’ (all ages-
P-value = 0.003), and ‘not significantly different’ (pre-Carboniferous ages- P-value = 0.113-0.165) at the 5% confidence level (Fig. 9a). This similarity is also illustrated in the approximation between SS-2, P-G-R-A-R and OMZ populations in the MDS diagrams (Figs. 9b, c).

K-S test results for the comparison between samples SS-2 and TM-3 indicate that they present ‘not significantly different’ detrital zircon populations (all ages- P-value = 0.399; pre-Carboniferous ages- P-value = 0.411) at the 5% confidence level (Fig. 9a). Furthermore, their cumulative probability curves are much closer (Fig. 8d): D-values are 0.195 (all ages) and 0.203 (pre-carboniferous ages) (Fig. 9a). The close relationship of the two detrital zircon populations suggests that the Toca da Moura volcano-sedimentary complex directly supplied sediment to the Santa Susana basin. However, the relationship described above does not extend to the entire Santa Susana basin since sample SS-1 presents a greater degree of similarity with the Cabrela detrital zircon population as regards the proximity between cumulative probability curves (Fig. 8d) and MDS diagrams (Figs. 9b, c).

In addition, Cabrela siliciclastic rocks are ‘significantly different’ at the 5% confidence level from sample TM-3 (P-values < 0.01) as regards the significant distance between them on the MDS diagram (Figs. 9b, c), and the significant distance between cumulative curves (Fig. 8d), with a D-value interval of 0.712-0.731 (Fig. 9a). The difference found in the detrital zircon populations suggests that Cabrela and Toca da Moura siliciclastic rocks probably derived from different sources.

As result of the K-S test and MDS analysis, the Horta da Torre Formation is ‘significantly different’ (Fig. 9a), and is clearly separate (Figs. 9b, c) from all the other detrital zircon populations, ruling out the possibility of it being a source for the Toca da Moura and Cabrela volcano-sedimentary complexes or Santa Susana Formation siliciclastic rocks.

6. Discussion

6.1. Chronostratigraphic framework

The geochronological data presented in the present study provide the basis for the first chronostratigraphic record for the Carboniferous basins of the Santa Susana-São Cristovão region (SW Iberia). Dating of silicic volcanic rocks interbedded in the Toca da Moura volcano-sedimentary complex constrain an interval of felsic magmatism to occurring from c. 335 Ma to 331 Ma (Visean; Fig. 6), complementing currently-available biostratigraphic information for Toca da Moura siliciclastic rocks (Pereira et al., 2006; Lopes et al., 2014). U-Pb ages of the youngest detrital zircon grains from the siliciclastic rocks of the Toca da Moura and Cabrela volcano-sedimentary complexes (TM-3 and CB, respectively; Fig. 8a) provide maximum age constraints for these marine deposits. Their maximum depositional ages (c. 351-348 Ma; Tournaisian) are slightly older than currently-available biostratigraphic ages (Pereira et al.,...
2006; Lopes et al., 2014), but provide confirmation that both marine deposits are broadly contemporaneous. Furthermore, the best estimate of the crystallization age of the Baleizão silicic intrusion provides a minimum age of c. 317 Ma (Bashkirian; Fig. 7a) for the intra-Carboniferous unconformity. Zircon extracted from a pebble of granite found in a Santa Susana conglomerate yielded a crystallization age of c. 303 Ma for plutonic rock (Fig. 7b). This age estimate overlaps the age interval of c. 305-303 Ma (i.e. the maximum depositional age range) obtained for the youngest population of detrital zircon grains from sandstone of the upper member (Dinis et al., 2018), complementing the currently-available biostratigraphic information for the Santa Susana Formation (Machado et al., 2012; Lopes et al., 2014). Given the findings described above, a stratigraphic interval of approximately 12-14 Ma can be established for the intra-Carboniferous unconformity, marking a change in depositional environment from marine to terrestrial in the OMZ. Basin-drainage and infill patterns most probably changed due to rapid uplift of the Variscan-Appalachian orogenic belt, active during the waning stages of Laurussia-Gondwana collision (i.e. Late Carboniferous).

6.2. Provenance and evolutionary model

An initial important finding based on the comparison of detrital zircon populations of Visean siliciclastic rocks from the Toca da Moura and Cabrela volcano-sedimentary complexes provides evidence that they derived from different sources. The TM-3 population presents 64% Precambrian detrital zircon grains, while the CB population contains only 10% (Fig. 8a). Toca da Moura siliciclastic rocks have a greater affinity with the Phyllite-Quartzite, Tercenas, Santa Iria and Represa formations (Fig. 9), indicating that detrital zircon populations were reproduced faithfully in SPZ and PLZ (Laurussian-type) sources. A contribution from the oldest siliciclastic sequences of PLZ (Pulo do Lobo, Atalaia, Gafo and Ribeira de Limas formations) and OMZ (Gondwanan-type) sources cannot be ruled out for sample TM-3 (Fig. 9). The number of Late-Middle Devonian zircon grains in sample TM-3 (6%) is smaller than that of the CB population (68%) (Fig. 8a), suggesting that Cabrela siliciclastic rocks were most likely derived largely from a Devonian source consistent with a limited contribution from recycled ancient rocks. This indicates that the origin of the Visean Toca da Moura and Cabrela basins is most likely more closely linked to sources located in the SPZ and PLZ (Laurussian-type) than in the OMZ (Gondwanan-type). The evidence in the Visean Toca da Moura basin for dissection of the inactive Devonian magmatic arc and the erosion of its plutonic roots, together with the recycling of the PLZ and SPZ Frasnian-Tournaisian siliciclastic sequences and OMZ basement rocks, differs from the evidence in the Cabrela basin. The significance of the involvement of distinct sources is that part of the region located on the boundary between the OMZ-PLZ and the SPZ (SW Iberia) was subjected to uplift while the remaining part underwent flexural subsidence. A
similar tectonic setting has been put forward as an explanation for differences in stratigraphy found in the Pedroches syn-orogenic basin located along the OMZ-Central Iberian Zone boundary (Armendáriz et al., 2008, and references therein) (Fig. 1). In the Visean, following the closure of the Rheic Ocean (i.e. subduction beneath the Laurussian margin up to the end of Devonian; Pérez-Cáceres et al., 2015; Pereira et al., 2017a, and references therein), sedimentation occurred simultaneously with igneous activity on both the Laurussian-side and the Gondwanan-side (Pereira et al., 2012b-Tecton). The upwelling of the asthenosphere and the underplating of mantle-derived magmas could have triggered partial melting of crustal materials and the intra-orogenic extension, creating the right conditions for the onset of gneiss domes (Pereira et al., 2009; Dias da Silva et al., 2018). The emplacement of voluminous magmatism with a composition typical of magmas produced, at convergent plate boundaries (Santos et al., 1990; Jesus et al., 2007, 2016; Pin et al., 2008; Lima et al., 2012; Pereira et al., 2007, 2015a; Moita et al., 2009, 2015), was simultaneously with flexural subsidence, marine sedimentation and volcanism in the Visean (Pereira et al., 2012b) (Fig. 10a). A factor which may explain this thermal anomaly is the subduction of an oceanic ridge beneath the OMZ (Gondwanan-side) during the initial closure of the PaleoTethys Ocean in the Carboniferous, whereas other regions of the Appalachian-Variscan orogenic belt experienced oblique collision and rapid uplift (Armendáriz et al., 2008; Pereira et al., in press), but as yet there is no consensus on this (Simancas et al., 2009; Cambeses et al., 2015).

A second significant finding is that detrital zircon populations from the Santa Susana Formation (samples SS-1 and SS-2) also show significant differences (Figs. 8 and 9). Basal conglomerate (sample SS-2) presents a greater percentage of Precambrian grains (64%) than uppermost sandstone (SS-1 sample; 28%), and presents a great degree of affinity with the detrital zircon population of sample TM-3. Sample SS-2 presents a great degree of similarity with the detrital zircon populations of overlying SS upper-member sandstones (samples SiSz-2 and SiSz-4; Dinis et al., 2018) sampled as part of the same stratigraphic profile. SS-2 and SS upper-age populations show a great degree of affinity (Fig. 9), suggesting that the two detrital zircon grains were mainly derived from the erosion of the Toca da Moura volcano-sedimentary complex, the Santa Iria and Represa formations (PLZ) and the Mira Formation (SPZ). However, regarding the detrital zircon grains with pre-Carboniferous ages, additional contributions from other PLZ (Pulo do Lobo, Atalaia, Gafo and Ribeira de Limas formations), SPZ (Brejeira, Phyllite-Quartzite, Tercenas and Ronquillo formations) and OMZ sources cannot be ruled out (Figs. 9a, c). The zircon age population of sample SS-1, which is distinct from the SS-2 population, presents a great degree of affinity with the CB population, suggesting lateral changes in sources during deposition of Santa Susana uppermost sandstones. The great degree of affinity of the SS-1, Cabrela volcano-sedimentary complex, with Mértola Formation detrital zircon populations suggests a close association between the two and a common source. Cabrela
and Mértola siliciclastic rocks may be regarded as the main source for sample SS-1 and an intermediate sediment repository as they are derived from the erosion of a Devonian source partially represented by the Cercal porphyries from the SPZ. As result of rapid uplift, the progressive erosion of the Devonian magmatic arc (including its plutonic roots), and that of PLZ, SPZ and OMZ rocks, is evidenced in the Santa Susana Formation. The volumetrically significant contribution of Carboniferous sources to the Santa Susana basin fill confirms derivation from the erosion of: i) Pyrite Belt volcanic rocks, and Phyllite-Quartzite, Tercenas, Mértola, Mira and Brejeira siliciclastic rocks (SPZ); ii) the Santa Iria and Represas formations (PLZ); iii) Gil Marquez granitic rocks and other plutons of the Sierra del Norte Batholith (SPZ and PLZ); iv) the Beja igneous complex, which includes the Baleizão porphyries (OMZ), and Évora and Pavia plutonic and high-grade metamorphic rocks (OMZ); and v) the Cabrela and Toca da Moura volcano-sedimentary complexes (OMZ) and Mértola turbidites (SPZ). U-Pb dating of magmatic zircon extracted from a pebble of granite (c. 303 Ma; Fig. 7b) found in a conglomerate of the Santa Susana Formation lower member suggests provenance from the direct erosion of Permo-Carboniferous plutons (i.e. original primary source), such as Santa Eulália-Monforte granitic and gabbro-dioritic rocks (OMZ). This c. 303-297 Ma calc-alkaline plutonic suite is coeval with the Nisa-Albuquerque and Los Pedroches batholiths, located on the OMZ-Central Iberian Zone boundary (Fig. 1), probably representing a magmatic arc related to the subduction of the Paleotethys Ocean (Pereira et al., 2015b, 2017b, in press). These Permo-Carboniferous plutons were emplaced at shallow crustal levels consistent with the low assimilation of country rocks and the sharp contacts, and therefore, they may have experienced denudation shortly after its crystallization without being required unrealistic uplift rates. In Kasimovian-Ghzelian, sedimentation probably occurred through the opening of the pull-apart terrestrial basin related to the movement of major strike-slip faults (i.e. Porto-Tomar fault zone, Machado et al., 2012, and references therein) during the waning stages of oblique continental collision between Laurussia and Gondwana, simultaneously with the progressive uplift of the Appalachian-Variscan orogenic belt (i.e. OMZ, PLZ and SPZ; Fig. 10b).

7. Conclusions

The main conclusions of this study are the following:

1. Visean marine deposition in the Santa Susana-São Cristovão region is constrained to the age interval of c. 335-331 Ma by the new U-Pb data for volcanic rocks intercalated within siliciclastic rocks of the Toca da Moura volcano-sedimentary complex.
2. U-Pb dating of the Baleizão porphyry provides a minimum age of c. 317 Ma (Bashkirian) for the overlying intra-Carboniferous unconformity.
3. Visean siliciclastic rocks from the Cabrela and Toca de Moura volcano-sedimentary complexes are derived from distinct sources, which probably include a Devonian continental
magmatic arc, and are likely to be more closely associated with the SPZ and PLZ (Laurussian-
type sources) than the OMZ (Gondwanan-type sources).

4. Terrestrial siliciclastic rocks from the Santa Susana Formation are probably the result of the
recycling of distinct sources associated with the SPZ, PLZ and OMZ.

5. The best estimate of crystallization of a granite pebble found in Santa Susana Formation
conglomerate yielded a maximum depositional age of ca. 303 Ma (Kasimovian-Gzhelian);
together with the youngest U-Pb ages (< c. 317 Ma) of detrital zircon grains, these findings
provide evidence of the denudation of primary crystalline sources during the rapid post-
accretion/collision uplift of the Variscan orogenic belt in SW Iberia (i.e. Gondwanan- and
Laurussian-type sources).

6. The intra-Carboniferous unconformity that separates the Toca da Moura volcano-complex
and the Baleizão porphyry from the Santa Susana Formation indicates a notable time interval of
approximately 12-14 Ma.

Acknowledgements

This work is a contribution to projects CGL2016-76438-P and PGC2018-096534-B-I00 (Spain),
the ICT’s Research Group 6- Lithosphere Dynamics (ICT-UID/GEO/04683/2019) and, IDL’s
Research Group 3- Solid Earth dynamics, hazards, and resources (Portuguese FCT). Í. Dias da
Silva acknowledges financial support by SYNTHERYS+ programme (2015-2016), FCT
postdoctoral grant SFRH/BPD/99550/2014 and FCT-project UID/GEO/50019/2019-IDL. This
is IBERSIMS publication number xx.

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Figure captions

Figure 1: A- Inset with location of SW Iberia in the Iberian Variscan belt with regional distribution of the main Paleozoic terranes: CIZ- Central Iberian Zone; CZ- Cantabrian Zone; GTMZ- Galicia-Trás-os-Montes Zone; OMZ- Ossa-Morena Zone; PLZ- Pulo do Lobo Zone; SPZ- South-Portuguese Zone and WALZ- West Asturian-Leonese Zone. B- Simplified Geological Map of SW Iberia showing the South-Portuguese, Pulo do Lobo and Ossa-Morena zones (Modified from Pereira et al. 2017a, 2019 and references therein; Quesada and Oliveira, 2019).

Figure 2: Simplified geological map and schematic stratigraphy of the Santa Susana-São Cristovão region (Ossa-Morena Zone; Modified from Gonçalves and Carvalhosa, 1984; Machado et al., 2012). Sampling locations of the Carboniferous sedimentary and igneous rocks used for geochronology are indicated with yellow stars.
Figure 3: Photographs of the Carboniferous igneous rocks of the Santa Susana-São Cristovão region: A- Baleizão porphyry intrusive contact (yellow arrow) with siliciclastic rocks of the Toca da Moura volcano-sedimentary complex; B- Baleizão porphyry; C-D- Rhyolitic tuffs of the Toca da Moura volcano-sedimentary complex; E- Volcanic breccia with fragments of siltstone (black) and rhyolite (yellow) at the base of the silicic tuffs from the Toca da Moura volcano-sedimentary complex; F- Pillow-lava of andesitic basalt-to-basalt intercalated in the siliciclastic rocks of the Toca da Moura volcano-sedimentary complex.

Figure 4: Photographs of the Carboniferous sedimentary rocks of the Santa Susana Formation lower member: A- View of dipping meter-thick beds of medium-coarse grained sandstone intercalated with conglomerate; B- Planar-bedded coarse-grained sandstone; C- Plant imprints in sandstone; D- Conglomerate with cobbles and pebbles of granite (G), quartzite (Q), silicic porphyry (SP) and mafic volcanic rock (M); E- Conglomerate with pebbles of rhyolite (R), phyllite (P), felsic tuff (T) and quartzite (Q).GG

Figure 5: Petrographic images of the Carboniferous sedimentary and igneous rocks of the Santa Susana-São Cristovão region: A- Rhyolitic-rhyodacitic tuff of the Toca da Moura volcano-sedimentary complex showing quartz and feldspar phenocrysts enclosed in ash matrix; B- Rhyolitic tuff showing flattened dark-brown millimeter-sized pumice and lithoclasts enclosed in ash matrix; C- Porphyritic texture of the Baleizão rhyodacite-rhyolite characterized by quartz, plagioclase, K-feldspar, biotite and amphibole phenocryst embedded in a fine-grained silicic matrix; D- Cobble of fine-grained granite showing graphic intergrowths of quartz and alkali feldspar, found in conglomerate from the Santa Susana Formation; E- Siltstone of the Toca da Moura volcano-sedimentary complex mostly composed of quartz grains and a few grains of plagioclase (P), tourmaline (T), and rock fragments (L); F- Siltstone of the Cabrela volcano-sedimentary complex showing fining upwards grading and a slump-fold; G-H, Sandstones from the Santa Susana Formation with high percentage of lithoclasts (L) and a few feldspar (F).

Figure 6: Concordia diagrams, weighted mean of $^{206}\text{Pb} / ^{238}\text{U}$ ages of analyzed zircon grains extracted from silicic tuffs of the Toca da Moura volcano-sedimentary complex.

Figure 7: Concordia diagrams, weighted mean of $^{206}\text{Pb} / ^{238}\text{U}$ ages of analyzed zircon grains of: A- the Baleizão porphyry and B- the cobble of granite found in conglomerate from the Santa Susana Formation.
Figure 8: Pie diagrams and Kernel Density Estimation (KDE) with U-Pb detrital-zircon ages of siliciclastic rocks from: A- the Toca da Moura (TM-3, this study) and Cabrela (CB: CBR-11, this study; and OM-200, Pereira et al., 2012a) volcano-sedimentary complexes, and B- the Santa Susana Formation (SS-1 and SS-2, this study; and SS Upper member, StSz2 and StSz4 from Dinis et al., 2018); C- U-Pb age cumulative frequency plots applied to the U-Pb ages (90-110% concordance) of detrital zircon grains from the Toca da Moura and Cabrela volcano-sedimentary complexes, and the Santa Susana Formation.

Figure 9: A- Results of the K-S (Kolmogorov-Smirnov) test and B- Multi-Dimensional Scaling diagrams (Vermeesch, 2018) applied to the U-Pb ages (90-110% concordance) of detrital zircon grains from the Toca da Moura (TM-3) and Cabrela (CB) volcano-sedimentary complexes, and the Santa Susana Formation (SS1, SS2, SS upper member), and different potential sources: OMZ (Linnemann et al. 2008; Pereira et al. 2008, 2012c), PLZ (Pereira et al. 2017; Pérez Cáceres et al. 2017), SPZ (Braid et al. 2011; Pereira et al., 2012a, 2014; Rodrigues et al. 2014). Abbreviations: MT- Mértola Formation; MR- Mira formation; BJ- Brejeira formation; PQ- TRC- Phyllite-Quartzite and Tercenas formations; SI-REP- Santa Iria and Represa formations; P-G-R-A-R- Pulo do Lobo, Gafo, Ribeira de Lima, Atalaia and Ronquillo formations; HT- Horta da Torre Formation.

Figure 10: Sketches showing inferred tectonic evolution and sedimentation recorded in SW Iberia Carboniferous stratigraphy during Laurussian-Gondwana oblique collision; A- Early Carboniferous; B- Late Carboniferous.
Figure 1

https://doi.org/10.5194/se-2020-26
Preprint. Discussion started: 11 March 2020
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Figure 2
Figure 3
Figure 4
Figure 5
Figure 6
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Figure 8
Figure 9

### A. Kolmogorov-Smirnov Test

#### All ages

| Test statistic | CB | SS1 | SS2 | SS3 | SS upper | MT | MR | BJ | PQ-TRC | SI-REP | P-G-R-A-R | HT | OMZ |
|----------------|----|-----|-----|-----|----------|----|----|----|--------|--------|-----------|----|-----|
| Test statistic | 0.717 | 0.797 | 0.819 | 0.797 | 0.819 | 0.797 | 0.819 | 0.797 | 0.819 | 0.797 | 0.819 | 0.797 | 0.819 |
| D-values using error in the CDF | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 | 0.184 |

#### Pre-Carboniferous ages

| Test statistic | CB | SS1 | SS2 | SS3 | SS upper | MT | MR | BJ | PQ-TRC | SI-REP | P-G-R-A-R | HT | OMZ |
|----------------|----|-----|-----|-----|----------|----|----|----|--------|--------|-----------|----|-----|
| Test statistic | 0.753 | 0.824 | 0.846 | 0.824 | 0.846 | 0.824 | 0.846 | 0.824 | 0.846 | 0.824 | 0.846 | 0.824 | 0.846 |
| D-values using error in the CDF | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 | 0.220 |

### MDS diagrams

#### All ages

- **Figure B**: MDS diagrams showing the relationship between different samples or categories, with labels denoting different groups or categories.

#### Pre-Carboniferous ages

- **Figure C**: MDS diagrams focusing on the pre-Carboniferous age group, with similar labels and groupings as Figure B.
Figure 10