

Maceral study to determine the desorbed gas content in the Upper Cretaceous coals of the Landázuri area, Middle Magdalena Valley Basin, Colombia

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Case study

Keywords: Middle Magdalena Valley, coal, petrology, maceral, gas, principal component analysis (PCA)

Posted Date: September 17th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-885749/v1>

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1 **Maceral study to determine the desorbed gas**
2 **content in the Upper Cretaceous coals of the**
3 **Landázuri area, Middle Magdalena Valley**
4 **Basin, Colombia**

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14

15 Abstract

16 For several decades, some coal petrographic properties have been proposed as important
17 parameters in the methane gas sorption processes. In this contribution, the petrographic variables
18 (Vitrinite Ratio, Inertinite Ratio, the petrographic indexes (Gelification Index, Groundwater Index,
19 Tissue Preservation Index, Vegetation Index, Vitrinite/Inertinite ratio, and the Vitrinite
20 Reflectance were evaluated according to the maceral preservation and were related with the
21 desorbed gas content. Twenty-five coal seams obtained from the drill cores of two wells in the
22 Landázuri Area-Valle Medio del Magdalena basin were analyzed. The coal samples were grouped
23 according to gas content using principal component analysis (PCA). The petrographic results were
24 analyzed by linear regression and multiple regression. The Medium Volatile Bituminous to Low
25 Volatile Bituminous coals from Landázuri 1 are twice as high in gas content that the High Volatile
26 Bituminous A to Medium Volatile Bituminous coals from Landázuri 2. The volume percentage
27 and the preservation degree macerals are related closely to the gas content and the pore's size
28 involved in the sorption process. The Inertinite is the maceral group related with the highest gas
29 content groups in Landázuri (600 SCF-Standard Cubic Feet/ton, 300 SCF/ton), while the other
30 groups show the correspondence with the vitrinite macerals. The syngenetic and diagenetic origin
31 of the pyrite contributes microporosity to the desorption process, while the pyrite epigenetic by its
32 size reduces it. The petrographic indexes reveal that the Upper Cretaceous coals were developed in
33 swampy or lacustrine continental basins- limnic facies.

34 *Keywords: Middle Magdalena Valley, coal, petrology, maceral, gas, principal*
35 *component analysis (PCA)*

36

37 **Introduction**

38 The Energy Resources group of the Servicio Geológico Colombiano has been
39 conducting Coal Bed Methane (CBM) studies since 2011 in five Colombian coal
40 areas. In 2016, total ash-free gas contents (37.54- 818.14 SCF/ton) were obtained
41 from coal seams of the Umir Formation-Upper Cretaceous of the Landázuri area-
42 Middle Magdalena Valley Basin. The resources potential was calculated as 360.47
43 BCF (Billion Cubic Feet) (Ortíz et al. 2016). Gas chromatography analysis
44 indicated that the methane gas percentage is between 41.90% and 92.50% of the
45 gas measured in the analyzed coal seams (Ortíz et al. 2016). The relationship
46 between methane gas accumulation mechanisms and coal composition has led to
47 the examination of certain measurements of interest in petrological analyses,
48 which act during the process of hydrocarbon sorption.

49 The largest volume of CBM resources worldwide is found in the countries of
50 Russia, China, United States, Canada, Australia, Indonesia, Poland, Germany, and
51 France, corresponding to 4000-7011 trillion cubic feet, of which 1060-1500
52 trillion cubic feet are considered recoverable. The United States has been the
53 leader in CBM production since the '90s with an upward trend, with 2008 being
54 the only decrease year; while China and Australia substantially increased their
55 own CBM production in 2010 and 2014, respectively, focusing on high-rank coals
56 and with interest in natural gas liquids (NGL) production. Thus, it is expected that
57 Australia will soon take the top spot on the global market, with India occupying
58 5th place (Mastalerz and Drobniak 2020).

59
60 In Colombia, coal is a primary source for electricity generation, being also the
61 largest producer in South America, with 1.46 exajoules (EJ)- 50 million tonnes in
62 2020 (BP 2021). Coal reserves reported during 2020 equaled 4554 million tonnes
63 of bituminous and anthracite coals (BP 2021). The Middle Magdalena Valley
64 basin has been in the sights of explorers because of its high hydrocarbon potential.
65 The first oil field discovered in the country is located. At the northeast of the basin
66 is located the Opón River- Landázuri coal area. Its potential is 201,012,049
67 million tonnes (Mt) of thermal coal and 137,156,216 Mt of metallurgical coal
68 from the Upper Cretaceous Umir Formation (Monroy and Sandoval 2014). In
69 terms of CBM reserve estimation, the analysis of two boreholes (Landázuri 1 and
70 Landázuri 2) allowed estimating a potential of 360.47 Billion Cubic Feet (BCF)

71 (Ortíz et al. 2016). The detailed petrological analyses are vital to know the coal
72 composition and the geological mechanisms of accumulation of coal-associated
73 gas in an area with high tectonic complexity.

74

75 Coal petrology studies began in Colombia from the year 2000 onwards in
76 different areas of the country (Castaño and Gómez 2001; Guatame and Sarmiento
77 2004; Mejía et al. 2006; Blandón 2007; Gómez and López 2017; Guo et al. 2018;
78 Akinyemi et al. 2020; Guatame and Rincón 2021, among others) mainly to
79 contribute to the knowledge of the petrographic composition and the definition of
80 the sedimentary environment.

81

82 Studies on CBM potential began in 1990 led by governmental entities and
83 universities, including, Agencia Nacional de Hidrocarburos- ANH, Ecopetrol-
84 Instituto Colombiano del Petróleo (ICP), Agencia Nacional de Minería (ANM),
85 Servicio Geológico Colombiano (SGC), Ministerio de Minas y Energía (MME),
86 Unidad de Planeación Minero Energética- UPME, Escuela de Administración,
87 Finanzas e Instituto Tecnológico-EAFIT Universidad Industrial de Santander-
88 UIS, Universidad Pedagógica y Tecnológica de Colombia- UPTC, and
89 Universidad Nacional de Colombia sede Medellín, among others (Mariño et al.
90 2015). In addition, the Drummond company stands out for carrying out the first
91 CBM exploitation project in the Cesar Ranchería basin. In the before-mentioned
92 studies, no petrological analysis of coal was performed, and in many of them, an
93 analysis of the influence of coal composition on methane gas content was not
94 rigorously elaborated. However, Mariño and Mojica (2014) carried out a
95 petrographic study in the Amagá basin on low-rank coals and concluded that the
96 vitrinite maceral group is the most influential component for gas adsorption and
97 that the mineral matter content evidences an inverse relationship concerning gas
98 content since it acts as a diluent.

99

100 Furthermore, under a significant number of studies from the sedimentary basins of
101 China, Australia, Canada, and the United States, crucial variables that influence
102 the sorption capacity of methane gas in coal seams to have been defined: the range
103 and burial depth (e.g., Laxminarayana and Crosdale 1999; Wang et al. 2011;
104 Bustin and Bustin 2016; Busch et al. 2019), the maceral composition (e.g. Yao et

105 al. 2009, Jian et al. 2015; Li et al. 2016; Hou et al. 2017), the pore size
106 distribution (micro, macro, mesoporous), and the specific surface area (e.g.,
107 Crosdale et al. 1998; Laxminarayana and Crosdale 1999; Chalmers and Bustin
108 2007; Busch and Gensterblum 2011; Li et al. 2014; Jian et al. 2015). According to
109 the notion that the most frequent maceral groups are Vitrinite and Inertinite, some
110 authors claim that macerals of the Vitrinite group present higher adsorption
111 capacity and higher desorption rate (e.g., Crosdale et al. 1998; Laxminarayana and
112 Crosdale 1999, among others). Others (Wang et al. 2011; Jian et al. 2015; Wang
113 et al. 2018; among others) express that it is the inertinitic macerals that have this
114 capacity. Regarding the mineral matter content resulting from the coal formation
115 processes, some authors indicate its relationship with the gas adsorption capacity
116 (e.g., Laxminarayana 1999; Moore 2012; Raharjo et al. 2018), with the desorption
117 rate (e.g., Crosdale et al. 1998; Li et al. 2017) and with the porosity contribution
118 in the sorption process (e.g., Bertrand 1989).

119

120 Colombian research on CBM potential has targeted different coal areas and has
121 yet to properly analyze the close relationship between the gas contents obtained
122 and the origin of the coal (Mariño and Mojica 2014). The research challenge is to
123 define which are the inherent physical properties of coals (rank, maceral
124 composition, mineral composition, facies, pore structure) involved in the higher or
125 lower gas content, which is necessary for the exploration of new economic
126 alternatives for coal deposits.

127

128 The main purpose of this study is to establish the relationship between the
129 measured gas content (SCF/ton) in the coal seams of the Landázuri 1 and
130 Landázuri 2 wells, and the maturity degree of the petrographic composition, and
131 in particular, the coal constituents that by their nature, preservation and the
132 occurrence mode, contribute to gas sorption. This research also aims to define a
133 methodology in exploratory studies of methane gas. Coal petrology is
134 implemented as a prognostic discipline that provides the necessary elements in the
135 CBM potential promising areas for the identification and prevention of gas-related
136 accidents in mining operations. Petrophysical studies of porosity and permeability
137 are not in the scope of this study.

138

139 **Geological setting**

140 The Landázuri 1 and 2 wells are located in the Armas syncline of the Río Opón-
141 Landázuri coal-bearing area (Landázuri- Vélez area) along the eastern margin of
142 the Middle Magdalena Valley basin (Fig.1). The area is affected by two regional
143 thrust fault systems resulting from the Mesozoic rifting of northern Colombia and
144 its subsequent reactivation during the Cenozoic for the time of the Andean
145 orogeny (e.g., Colleta et al. 1990; Dengo and Covey 1993; Cooper et al. 1995, for
146 more references see Sarmiento et al. 2015). The oblique compressional stress
147 generated structures and reactivated transpressive faults generating second-order
148 folds oblique to these main faults (Gómez et al. 2008 in Ortíz et al. 2016).

149

150 In the research area, the La Salina fault system corresponds to a reverse system
151 with westward vergence spans from south to north in the western flank of the De
152 Armas syncline putting in contact Maastrichtian rocks with those dated Lower to
153 Middle Oligocene (Sarmiento et al. 2015; Ortíz et al. 2016). The Landázuri Fault
154 system with westward vergence corresponds to a thrust scale of the La Salina
155 Fault, located on the eastern flank of the syncline where Turonian rocks overlying
156 Maastrichtian rocks. (Ortíz et al. 2016). The Armas syncline corresponds to a
157 broad and asymmetric fold with southeast-northwest direction and widening on
158 the eastern flank (Ortíz et al. 2016).

159

160 In the Upper Cretaceous rocks, the roughly 1400 meters thick Umir formation
161 (Gómez et al. 2008 in Ortíz et al. 2016; Sarmiento et al. 2015) was deposited in a
162 restricted marine environment with the development of marshy areas that
163 accumulate organic matter for the subsequent production of economically
164 exploitable coal seams (Sarmiento et al. 2015). The unit consists of three levels. In
165 the Landázuri area the thickness is tectonically affected and the lower level of the
166 Umir Formation or La Renta Formation- (Terraza 2020) is not evident. The
167 middle level is 400 meters thick composed of a gray claystone succession with
168 gray to light brown silty claystone intercalations and sporadic levels of very fine
169 to fine-grained arenite. The segment has coal seams with variable thicknesses
170 ranging from a few centimeters to 3 meters. The upper level is 400 meters thick
171 and consists of a succession of light brown, intercalated with gray clayey
172 siltstones and coal seams of fine-grained arenite (Ortíz et al. 2016).

173 The Landázuri 1 well is located on the eastern flank of the De Armas syncline in
174 the southern sector, crossing the upper and middle levels of the Umir Formation.
175 Fourteen coal seams numbered from base to top (M3- M23) were analyzed
176 according to the stratigraphic position with variable thicknesses ranging from 0.40
177 m to 2.63m (Ortíz et al. 2016). The Landázuri 2 well is located northwest on the
178 western flank of the syncline. On the middle level, eleven coal seams were
179 identified (M2- M18) with thicknesses varying from 0.55 m to 2.38 (Ortíz et al.
180 2016) (Fig. 1 and 2).

181

182 **Experimental methods**

183 **Gas measurement method**

184 The gas desorption measurement of the cores was performed through the direct
185 canister method, as desorbed and residual gas as a function of time and under
186 pressure and temperature conditions that simulate the reservoir characteristics in a
187 laboratory environment (Ortíz et al. 2016).

188

189 The coal degassing (lost gas) on the drilling cores consisted of the water
190 displacement measurement in a volumetric column. After 24 hours, the
191 measurement of desorbed gas was started in hermetically sealed canisters with in
192 situ pressure and temperature control or calculated with the geothermal gradient.
193 The absorbed gas by the coal matrix was calculated from the residual gas in
194 pulverized samples (60 mesh) in a container for gas mixture or in a hermetically
195 sealed ball mill connected to a manometer. The measurements obtained were
196 processed using the TerraGas software (<https://terrasolid.com/product/tgas/>),
197 which performs pressure, temperature, and gas volume corrections, measured
198 from the sample weight, canister volume, volume of inert material introduced, and
199 density (ISO Standard 17892-2:2014). The cumulative gas desorption curve points
200 out the intervals of lost, measured and residual gas by plotting the cumulative gas
201 content vs time (ft/ton Vs $\sqrt{\text{hours}}$) (Fig. 3). The ash correction was performed
202 finally obtaining the ash-free total gas content. (Ortíz et al. 2016). Gas
203 chromatography was used to determine the percentage of gas mixture and
204 methane gas in the measured contents of each sample (Ortíz et al. 2016).

205

206 **Petrographic methods**

207 The reflectance values (Ro) were obtained by professionals from the Servicio
208 Geológico Colombiano's Laboratory of Characterization, Processing and
209 Research of Coal and Energetic Materials, according to the ASTM D2798-05
210 standard. The identification and classification of the macerals was carried out
211 according to Standard D2799-05 with the reading of 500 points in each specimen
212 and its corresponding repeatability. A Leitz Ortholux II POLMK microscope was
213 used with a magnification of 400x, a dot counter, and a mechanical stage that
214 advances with fixed increments. The macerals classification used the
215 nomenclature compiled by ICCP (1998, 2001) and the Pickel et al. (2017)
216 nomenclature for the Liptinite group. The macerals of the Vitrinite and Inertinite
217 groups were differentiated according to their degree of preservation (Lamberson
218 and Bustin 1993). Structured Vitrinite is composed of the Telovitrinite and
219 Gelovitrinite subgroup and the degraded Vitrinite is constituted of macerals of the
220 Detrovitrinite subgroup. Preserved Fusinite and Semifusinite were counted as
221 preserved inert, and the other macerals group make up the degraded inerts
222 subgroup. The mineral matter was quantified as clay, pyrite, and others. The
223 Pyrite classification was according to its formation process in coal as syngeneic,
224 diagenetic and epigenetic. The coal facies were obtained from the application of
225 petrographic indices proposed by Diessel (1986) and Calder et al. (1991).

226

227 **Statistical methods**

228 To avoid the analyst's subjectivity when classifying coal seams groups, cluster
229 analysis was used, based on K-Means Clustering (Hartigan and Wong 1979). This
230 procedure attempts to discriminate groups of relatively homogeneous cases, based
231 on certain selected properties. In our particular case, the following variables were
232 used: RV, RI, GI, GWI, TPI, VI, V/I. Pseudocode written in R was used, which
233 allows a large number of cases to be considered. However, it is often the case that
234 the algorithm requires the user to specify the number of clusters. For this, the
235 initial number of the clusters was obtained from the Elbow Method (Lloyd 1982).
236 In the algorithm, the initial centroids of the clusters can be specified, and the

237 cluster centroids are updated iteratively. The algorithm used also allows automatic
238 selection of the number of clusters based on Bayesian information criteria
239 (Schwartz 1978). It can also store cluster labels for each measurement, distance
240 information, and the centers of the final clusters. The relative size of the statistics
241 provides information about the contribution of each variable to the separation of
242 the clusters. Once the classification is performed; the results are presented in the
243 form of dendograms. The R correlation factor is analyzed as a linear regression
244 product from the petrographic variables concerning the gas content in the defined
245 groups. Then, for each group, a Pearson correlation coefficient analysis was
246 carried out (Kendall and Stuart 1973), which allowed us to determine different
247 relationships between variables pairs. The most significant variables from this
248 independent study were compared statistically with each petrographic variable.
249 Finally, the stepwise multiple regression method was applied, in which the
250 contribution of each predictor variable to the response variable was analyzed. The
251 algorithm determines which are the most important predictor variables for each
252 one of the analyzed wells.

253

254 **Results and discussion**

255 **Gas content**

256 The ash-free gas total content results (Ortíz et al. 2016) in the twenty-five coal
257 seams analyzed ranges between 37.54- 818.14 SCF /ton. The lowest value
258 corresponds to M-15 from the Landázuri 2 well and the highest value measured at
259 M-3 from the Landázuri 1 well.

260

261 **Petrographic analysis**

262 To the southeast of the De Armas syncline (see Fig. 1), the average reflectance of
263 the Vitrinite ranges between 1.22- 1.86% MVB-LVB for Landázuri 1. The
264 measured gas content fluctuates between 80.66 -818.14 SCF/ton, where 78.90-
265 91% of the values obtained correspond to methane gas. To the northwest of the
266 structure, coals are classified as HVBA- MVB for Landázuri 2 with values
267 between 0.9 and 1.26% reflectance. The measured gas content is in the range

268 37.54 -484.88 SCF/ton, where 41.90- 92.50% of the obtained values correspond to
269 methane gas (Ortiz et al. 2016; see Tables 1 and 2).

270

271 After having undergone the desorption process, the macerals showed numerous
272 empty spaces of irregular size and shape. As shown in Tables 1 and 2, the
273 Vitrinite content ranges between 65.71% and 94.03% in the analyzed samples.
274 Likewise, the highest values correspond to the subgroup Detrovitrinite
275 (Colodetrinite, Vitrodetrinite) and Gelovitrinite (Gelinite Colodetrinite involves
276 different macerals such as Inertodetrinite and is observed partially fusinitized and
277 devolatized (Fig. 5c, 5f, 5k, 5l, 5n, 6c, 6f, 6h). Gelinite is of a homogeneous
278 aspect and is occasionally fractured (Fig. 5e, 6d), Vitrodetrinite is observed as
279 angular fragments and is concentrated in the edges of the Vitrinite macerals (Fig.
280 5j, 6e).

281

282 The macerals content of the Inertinite group varies between 5.97-34.07% in the
283 analyzed coal seams. Inertodetrinite is the most abundant maceral and is observed
284 contained in Colodetrinite mainly (Fig. 5f, 5m, 6f, 6i). Fusinite and Semifusinite
285 were quantified as preserved macerals-preserved cell structure and degraded-no
286 cell structure (Fig. 5e, 5m, 54n, 5o, 6h, 6k). The macerals empty spaces may be
287 empty or with mineral matter filling (Fig. 5e, 5g, 5i, 5b, 6g, 6h, 6j, 6k). The
288 Liptinite content is very low or zero, and therefore, the maceral group was not
289 analyzed.

290

291 The mineral matter comprises clay filling, replacing macerals (Fig. 5a, 5d, 6a, 6c)
292 and pyrite, with values between 3.60 to 30.00%, obtaining the highest values for
293 Landázuri-1 (see Tables 1 and 2). In the coal seams analyzed, the pyrite was
294 characterized according to morphology into framboidal, granular, massive, and
295 crystalline. It was observed replacing macerals and filling voids and fractures
296 (Fig. 5b, 5g, 5h, 5i, 6b, 6j).

297

298 Correlations between macerals of Vitrinite and Inertinite groups, and the
299 petrographic indexes (GI, TPI, VI, GWI, and Vitrinite- VI radius) are listed in
300 Tables 3 and 4.

301

302 **Group selection**

303 The Elbow plots shown in Figures 8 A and B allows us to discriminate seams
304 groups for each well. Three seams' groups from the Landázuri 1 well show that
305 the quadratic distance between the elements that comprise them is less than
306 20,000. While Landázuri 2 well has a distance between two and three seams'
307 groups in the range between 30,000 and 50,000. Although, for both wells with 4
308 and 5 groups, the distance is minimized the samples number within each was
309 taken into account. The Landázuri 1 well consists of fourteen core samples, while
310 the Landázuri 2 well comprises 11 samples. Accepting groups of 4 or 5 would
311 imply that they would be formed by 1 or 2 samples and, in that case, the
312 correlations between the variables would not make any geological sense, since
313 any straight line could be adjusted by linear regression of two measurements.
314 Thus, we decided to keep the 3 groups for Landázuri 1 and 2 groups for Landázuri
315 2 (Fig. 7). The dendograms produced using PCA (Principal Component Analysis)
316 analysis suggest that the samples integrate each group according to the measured
317 gas content (Fig. 8).

318

319 **Linear regression analysis**

320 *Relationship between measured gas volume and Vitrinite Reflectance*

321 The coal rank has been defined as an important factor in the maceral composition
322 variation and, therefore, in the gas storage capacity (e.g., Crosdale et al. 1998,
323 2017; Keshavarz 2017). Other authors deduced that a higher coal rank indicates a
324 greater depth, and as the Vitrinite percentage increases the gas content is higher
325 (Bustin and Clarkson 1998, among others). In the Landázuri coals, the reflectance
326 analysis reveals that the gas content does not increase proportionally with rank,
327 nor with depth, taking into account that the highest reflectance values are not
328 found in the lowest stratigraphic position (see Fig. 9 a,c).

329 It is evident that the middle-to-high range coals- (MVB- LVB) from the Landázuri
330 1 well, duplicate in gas content the lower-range coals (HVB - MVB) from the
331 Landázuri 2 well. The gas content (< 600 SCF / ton- Landázuri 1 and <300-
332 Landázuri 2) have no relationship with Vitrinite Reflectance, while the groups

333 with higher gas content present a correlation factor $R^2 = 0.74$ for Landázuri 1 and
334 0.99 for Landázuri 2 (Fig. 9 b, d).

335

336 *Relationship between measured gas volume and the Vitrinite Group*

337 The relationship between the Vitrinite percentage and the gas content indicates as
338 the Vitrinite content increases, the gas adsorption capacity decreases, showing a
339 negative slope in the correlation graph (Hackley et al. 2000; Mares and Moore
340 2008). The above applies to the groups defined with the higher measured gas
341 content (>600- Landázuri 1 and >300- Landázuri 2). The other groups show a
342 positive correlation, which indicates that as the percentage of Vitrinite the
343 measured gas content also increases. The coal rank inclusion suggests that the
344 Vitrinite content does not influence gas adsorption in the BMV-BBV rank coals
345 (Laxminarayana and Crosdale 1999). That is the case for the Landázuri coals,
346 with > 600 SCF/ton and > 300 SCF/ton gas content, the opposing relationship
347 occurs in lower gas content coals. (see Fig. 10 a, b).

348

349 The dependence between structured (Telovitrinite, Gelovitrinite), and degraded
350 (Detrovitrinite) macerals defines the Vitrinite radius. Fig. 10 shows the correlation
351 with positive character in the medium to high-rank Landázuri 1 coals with a gas
352 content measured between 200-800 SCF/ton. The VR is irrelevant in <200 SCF /
353 ton gas content coals because of its zero tendency. The coals of the Landázuri 2
354 well have a positive correlation with the gas content reflecting the Vitrinite
355 structured macerals importance. (See Fig. 10 c, d; Tables 3 and 4).

356

357 *Relation between measured gas content and the Inertinite Group*

358 The correlation of the Inertinite percentage with the gas content suggests a
359 positive influence in the groups with higher gas content (>600 and >300 SCF/ton).
360 The remaining groups indicate a correlation with a negative trend. (See Fig. 11;
361 Table 1 to 4).

362 The IR has merit in the defined coals groups, specifically in the >600 SCF/ton gas
363 content -Landázuri 1, where the degraded Inertinite is higher than the structured
364 Inertinite. (See Fig. 11a, b; Table 1 to 4).

365

366 The Inertinite cellular structure is an important factor in methane gas enrichment
367 in the defined groups with higher gas content. It is contrary to what was observed
368 with the percentage of Vitrinite with a high degree of metamorphism that does not
369 allow filling the pores with gas. (e.g., Mares and Moore 2008; Wang 2018).
370 Although an inverse relationship of the Inertinite radius is observed in the > 600
371 SCF/ton group, the pore size distribution and its role in the gas desorption process
372 in both structured and the degraded inerts remain significant (see Fig. 11c, d).

373

374 *Relationship between the measured gas content and the mineral matter*
375 *percentage*

376 The relationships between the mineral matter content with the measured gas
377 content do not show correlation or tend to zero, except in the group <300 SCF/ton
378 in Landázuri 2 (see Fig. 12). The pyrite is observed as euhedral and anhedral,
379 framboidal grains or crystals, filling pores and voids, as an indicator of a
380 syngenetic process (Raharjo 2018; Dai et al. 2020). The early syngenetic and
381 diagenetic origin is related to a higher gas content in the coals of Landázuri 1.
382 According to Bertrand (1989), this condition is due to the contribution of the
383 microporosity of the mineral matter after the gelation process. The different forms
384 of pyrite concentration are shown in Fig. 13. We discriminated that epigenetic
385 pyrite concerns intermediate to low gas contents as occurs in coal seams: M17 and
386 M2 in Landázuri 2, this reduction in methane gas sorption, according to Raharjo
387 (2018) could be due to the large size and area covered by pyrite, which, according
388 to Laxminarayana (1999), and Warwick et al. (2008) reduce gas sorption by a
389 retention process.

390

391 *Relationship between measured gas content and the coal facies*

392 The calculated indices were: TPI- Tissue Preservation Index, GI- Gelation Index,
393 VI- Vegetation Index, GWI- Groundwater Index, and V/I radius. They are the
394 water table variations concerning organic matter preservation and are calculated
395 as follows:

396

397 TPI = Telinite + Colotelinite + Semifusinite + Fusinite / Colodetrinite+ Macrinite
398 + Inertodetrinite

399 GI= Vitrinite + Macrinite / Semifusinite + Fusinite + Inertodetrinite

400 GWI = Gelinite + Corpogelinite + Minerals (clay, quartz) + Vitrodetrinite /
401 Telinite + Telocolinite + Colodetrinite

402 VI = Colotelinite + Fusinite + Semifusinite + Suberinite + Resinite / Colodetrinite
403 + Inertodetrinite + Alginite + Liptodetrinite + Sporinite + Cutinite

404

405 The index that reveals the degree of persistence of wet and dry conditions is the
406 GI (see Fig. 14e, f); and the GWI and the Vitrinite/Inertinite ratio (V/I) represent
407 the degree of gelation of the fabrics according to the water and pH contributions
408 (see Fig. 14c, d, i, j). These indices show the same behavior as the percentage of
409 Vitrinite concerning the gas content illustrated in Fig. 10a, b. The GI value
410 exception is the Landázuri 2 well with >300 SCF/ton content, with a very low
411 Telovitrinite and Colodetrinite content; therefore, the index acquires a high value.
412 On the other hand, the TPI index as an indicator of the degree of humification of
413 organic matter macerals (see Fig. 14a, b) and the VI index (see Fig. 14g, h) show
414 the contrast of macerals of forest affinity with herbaceous marginal, indicating the
415 same trend as that of the VR in Fig. 10c, d.

416

417 The Calder et al. (1991) and Diessel (1986) models indicate that the organic
418 matter in Landázuri was transformed in a limnic environment (marshy or
419 lacustrine continental basins) that fulfills the Vitrinite> Inertinite and Degraded
420 Vitrinite> Structured Vitrinite condition (Fig. 15 and 16). The coal facies
421 variation is not evident in the same geological formation, as the seams are from
422 the same well.

423 **Pearson's analysis**

424 The relationships between pairs of variables are plotted as Pearson correlograms,
425 these summarized the linear regression analysis, highlighting the inertinitic
426 macerals contribution in the group of each well with higher desorbed gas content
427 and of Vitrinite (V) and V/I radius in the coals categorized with medium to low

428 gas content (see Fig. 17). For these correlograms, only variables that have a
429 relationship were plotted using a p-value of 0.25. Additionally, the correlograms
430 allow us to corroborate other linear relationships between variables that had not
431 been considered before and to identify their influence on the response variable, as
432 is the case of gas content.

433

434 **Multiple regression analysis**

435 The analysis was performed considering the variable Gas V (Gas Volume) as the
436 response variable and the variables: Ro, StrV, StrI, Gas V, %V, and %I as the
437 predictor variables.

438

439 In Tables 5 and 6, a summary of the multiple linear regression statistics is
440 presented for the initial and final models of the Landazuri-1 well. According to
441 the Table 5 none of the five predictor variables (Ro, Str I, Str V, %V and %I)
442 would be significant in the calculation of gas volume. The closest is Str V with a
443 probability of 0.133. The adjusted regression coefficient is 0.05362 and p-value=
444 0.3984. Once the stepwise selection procedure is applied, Table 6 shows that the
445 Str V variable is significant assuming a reliability level of 99%, the adjusted R2=
446 0.229 increases, while the p-value diminishes to 0.08328, which imply that the
447 two predictors' variables Str I and Str V are significant to reproduce the gas
448 volume.

449 Considering the same five predictor variables previously mentioned, in Table 7
450 we summarized the multiple linear regression results for the Landazuri-2 well.
451 According to this Table, only Str V is a significant variable with a probability of
452 0.0998 for the gas volume estimation. However, Ro and Str I are other variables
453 that could have a significant effect on gas volume, with probabilities of 0.2411
454 and 0.2235, respectively. After the application of the stepwise method adjusted R2
455 correlation coefficient shows an improvement with respect to the initial model,
456 evolving from 0.6991 to 0.7508. Similarly, the p-value of the initial model
457 improves from 0.04023 to 0.004795 in the final model. Thus, Ro, Str I, and Str V
458 represent significant predictors assuming a confidence level of 99%. (see Table 8).
459 Fig. 18 shows in blue those predictor variables that have a positive influence with
460 the gas content. The analysis highlights the Reflectance Vitrinite importance and

461 the structured macerals content in the coal seams related to the gas content, which
462 is a consequence of the coal formation history.

463

464 **Conclusions**

465 The measured gas volumes (SCF/ton) in the rank HVBA- MVB Landázuri 2 coals
466 fluctuate between 37.54 to 484.8, while rank MVB- LVB Landázuri 1 coals
467 doubles it, reaching values of 818.14 SCF/ton. The definition of the groups
468 according to the measured gas content allows highlighting the relationship of the
469 maceral composition with the pore size (micropore, macropore, mesopore), and
470 the coal rank.

471

472 The petrographic variables analyzed to reveal that the groups with the highest gas
473 content (> 600 SCF / ton- MVB, LVB-Landázuri 1; > 300 SCF / ton- HVBA,
474 MVB-Landázuri 2) exhibit higher content of inertinite (macropore, mesopore),
475 while the medium to low gas content is directly related to the content of vitrinite
476 (micropore) emphasizing structured macerals (see Fig. 19).

477

478 Further, mineral matter contributes microporosity about gas content showing
479 positive correlation except in coals >300 SCF/ton from Landázuri 2. The pyrite
480 syngenetic and early diagenetic is related to higher gas content, while epigenetic
481 pyrite concentration covers the greater surface area and is related to intermediate
482 to low measured gas contents.

483

484 The maceral relationship in the definition of the petrographic indices reveals
485 limnic facies of continental swampy or lacustrine basins. The GI, GWI, and V/I
486 indices present the same behavior as the Vitrinite content in each group analyzed,
487 except for the high GI value in the group with >300 in Landázuri 2 well, as the
488 result of the low Telovitrinite and Colodetrinite content. The TPI and VI indices
489 are equivalent to the Vitrinite radius.

490

491 Acknowledgments

492 The authors would like to thank the geological engineers Claudia Duarte, Luis Fernando Ortiz,
493 Fernando Andres Parra of the Energetics Group - Servicio Geológico Colombiano, for the detailed
494 field work, drilling and desorption analysis of the coal seams; the group of professionals of the
495 Laboratory of Characterization, Processing and Research of Coal and Energetic Materials -
496 Servicio Geológico Colombiano, for the reflectance analysis of the selected samples. Mauricio
497 Bermúdez thanks the UPTC SGI-DIN-3104 Project.

498

499 **Declarations**

500

501 **Funding** This research is part of the Exploration in Energy Resources Project of
502 the Servicio Geológico Colombiano.

503

504 **Availability of data and materials** Applicable.

505

506 **Conflict of interest:** The authors have no conflicts of interest to declare that are
507 relevant to the content of this article.

508

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727

728 **FIGURES CAPTIONS**

729 **Fig. 1** Geological map of the Landázuri- Vélez area- Middle Magdalena Valley Basin. Source:
730 (Ortíz et al. 2016)

731

732 **Fig. 2** Generalized stratigraphic column of the Upper Cretaceous- Middle Magdalena Valley Basin
733 and Coal seams distribution from Landázuri 1 and 2 wells. Source: (Ortíz et al. 2016; Etayo-Serna
734 2019)

735 **Fig. 3** Gas desorption curve (Ortíz et al. 2016)
736

737 **Fig. 4** Ash-free gas content (SCF/ton) of coal seams from Landázuri 1 and Landázuri 2 wells
738 (Ortíz et al. 2016)
739

740 **Fig. 5** Coal seams photomicrographs of the Landázuri 1 well after desorption gas process. a. Clay
741 filling inert's empty spaces -M23, b. Framboidal pyrite -M21, c. Devolatilized Colodetrinite -M20,
742 d. Clay mineral matter -M17, e. Fractured Vitrinite and structured Fusinite -M14, f. Inertodetrinite
743 in devolatilized Colodetrinite -M13, g. Framboidal pyrite in Semifusinite -M12, h. Mineral matter -
744 M11, i. Pyrite -M10, j. Vitrodetrinite, k. Colodetrinite -M7, l. Devolatilized Vitrinite -M6, m.
745 Preserved Fusinite and Inertodetrinite- M5, n. Vitrinite and inerts after desorption -M4, o.
746 Degraded inerts - M3
747

748 **Fig. 6** Coal seams photomicrographs of the Landázuri 2 well after desorption gas process. a. Clay
749 minerals -M18, b. Pyrite filling Inertinite's spaces -M17, c. Devolatilized Vitrinite - M16, d. Clay
750 minerals in fractured Vitrinite -M15, e. Colotelinite and Vitrodetrinite -M14, f. Inert in
751 Colodetrinite -M11, g. Semifusinite's spaces filled with mineral matter -M10, h. Empty spaces in
752 Colodetrinite and Semifusinite - M9, i. Inertodetrinite in Colodetrinite -M5, j. Pyrite in Fusinite -
753 M3, k. Fusinite -M2
754

755 **Fig. 7** Elbow-plots for (A) Landázuri 1and (B) Landázuri 2 wells
756

757 **Fig. 8** Dendograms showing results of cluster analysis (A) Landázuri-1 (B) Landázuri-2 wells
758

759 **Fig. 9** Coal rank (Ro) relation with depth and gas content measured (%Ro) (a, c) Landázuri-1 well.
760 (b,d) Landázuri-2 well

761 HVBA- High Volatile Bituminous A, MVB- Medium Volatile Bituminous, - LVB Low Volatile
762 Bituminous.
763

764 **Fig. 10** Relationship between Vitrinite percentage (fmmb) and the Vitrinite Radius with the gas
765 content (SCF/ton) (a, c) Landázuri-1 well, (b,d) Landázuri-2 well
766

767 **Fig. 11** Relationship between Inertinite content (fmmb) and the Inertinite Radius with the gas
768 content (scf/ton) (a, c) Landázuri-1 well, (b, d) Landázuri-2 well
769

770 **Fig. 12** Relationship between mineral matter and the gas content (SCF/ton) (A). Landázuri 1 (B)
771 Landázuri 2
772

773 **Fig. 13** Microphotographs a. Clay minerals in M5- Landázuri-1, b. Clay minerals and pyrite -M17-
774 Landázuri-2, c. Clay and others -M21- Landázuri-1, d. Framboidal pyrite -M17- Landázuri-2, e.
775 Framboidal pyrite -M17-Landázuri-2, f. Framboidal pyrite -M7-Landázuri-1, g. Framboidal pyrite
776 -M7-Landázuri-2, h. Syngenetic-diagenetic pyrite -M3-Landázuri-2, i. Epigenetic pyrite -M17-
777 Landázuri-2, j. Epigenetic pyrite -M2-Landázuri-2, k. Epigenetic pyrite -M17-Landázuri-2, l.
778 Syngenetic framboidal pyrite -M16-Landázuri-2, m. Diagenetic pyrite -M17-Landázuri-1, n.
779 Diagenetic pyrite -M4-Landázuri-1, o. Diagenetic pyrite -M10-Landázuri-1, p. Late diagenetic
780 pyrite? -M17-Landázuri-2

781 **Fig. 14** Relationships between gas content (SCF/ton) and petrographic indexes (TPI, GWI, GI, VI,
782 and V/I)
783

784 **Fig. 15** Coal facies (GI, TPI) Source: Diessel (1986) and modified by Kalkreuth et al. 1991. a.
785 Landázuri 1. b. Landázuri 2 A. Vitrinite> Inertinite, Degraded Vitrinite> Structured Vitrinite B.
786 Vitrinite> Structured Inertinite, Vitrinite> Degraded Vitrinite C. Inertinite> Vitrinite
787 Inertodetrinite> Semifusinite- Fusinite D. Inertinite> Vitrinite Semifusinite+Fusinite>
788 Inertodetrinite
789

790 **Fig. 16** Coal facies (GWI, VI). a. Landázuri 1 b. Landázuri 2 (Modified from Calder et al. 1991)
791

792 **Fig. 17** Pearson correlograms showing relationships between pairs of variables for Landázuri 1
793 and Landázuri-2

794 Ro- Average Vitrinite Reflectance, V fmmb - Vitrinite free mineral matter, I fmmb- Inertinite free
795 mineral matter, mm- mineral matter, V/I- Vitrinite/Inertinite radius, Deg V- Degraded Vitrinite,
796 Str I - Structured Inertinite, Deg I - Degraded Inertinite, Str V- Structured Vitrinite, RV- Vitrinite
797 Radius, IR- Inertinite Radius, Gas V- Total gas volume measured SCF/ton
798

799 **Fig. 18** Behavior analysis of predictor variables (Str V, Str I, %V, %I, Ro) versus response
800 variable (Gas V) for the wells: a. Landázuri 1 and b. Landázuri 2
801

802 **Fig. 19** Synthesis of the importance of maceral groups and reflectance for the measured gas
803 content

804 TABLES CAPTIONS

805 **Table 1.** Mean Vitrinite reflectance, measured gas volume, and maceral composition of the coal
806 seams - Landázuri 1 well

807 **Table 2.** Mean Vitrinite reflectance, measured gas volume, and maceral composition of the coal
808 seams- Landázuri 2 well

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810 **Table 3.** Relationships between maceral content and petrographic indexes for coal seams of the
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815 **Table 5.** Summary of multiple linear regression for initial model of the Landazuri-1 well

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818

819 **Table 7.** Summary of multiple linear regression for initial models of the Landazuri-2 well

820

821 **Table 8.** Summary of multiple linear regression for final model of the Landazuri-2 well

Figures

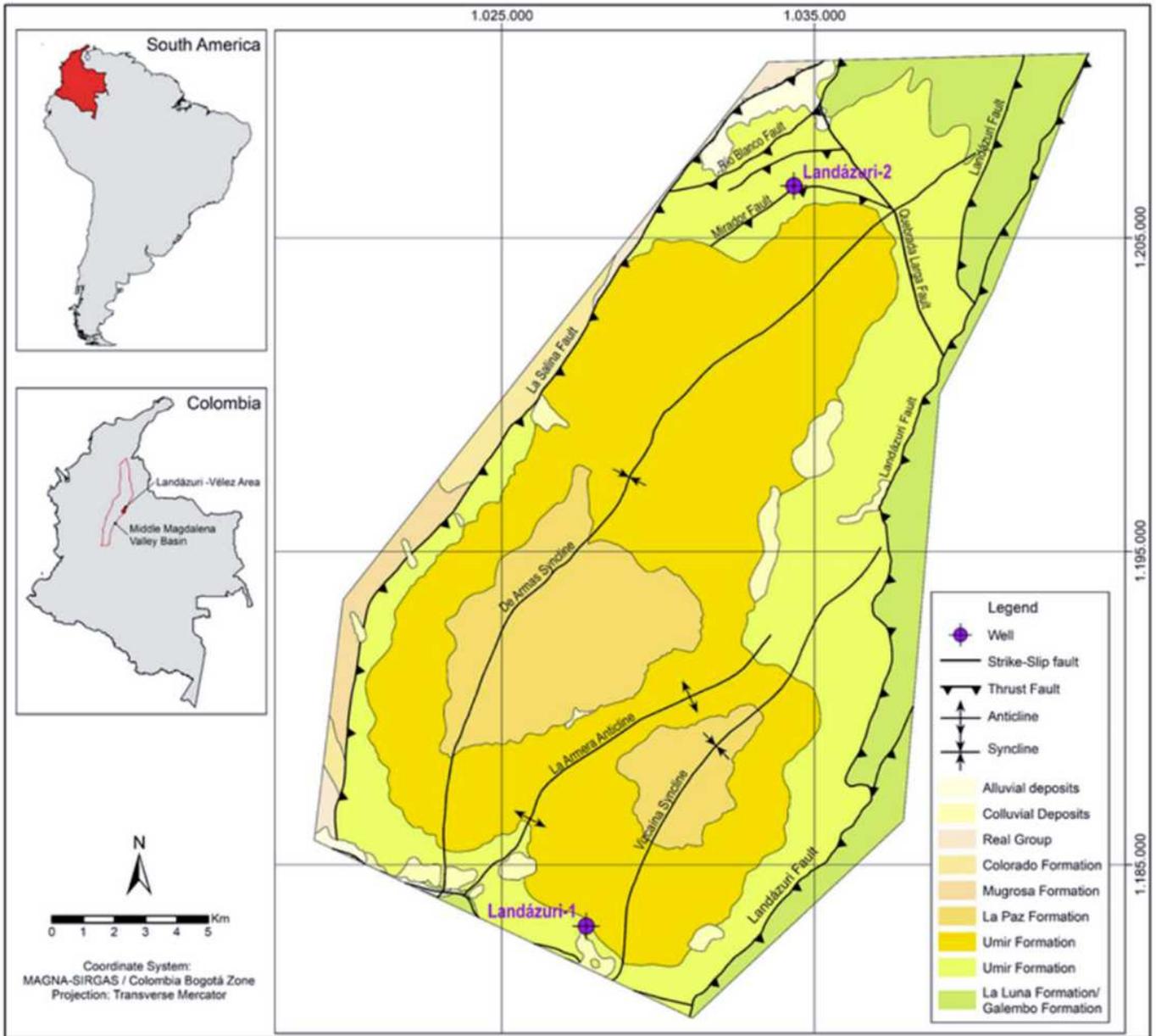


Figure 1

Geological map of the Landázuri- Vélez area- Middle Magdalena Valley Basin. Source: (Ortíz et al., 2016)

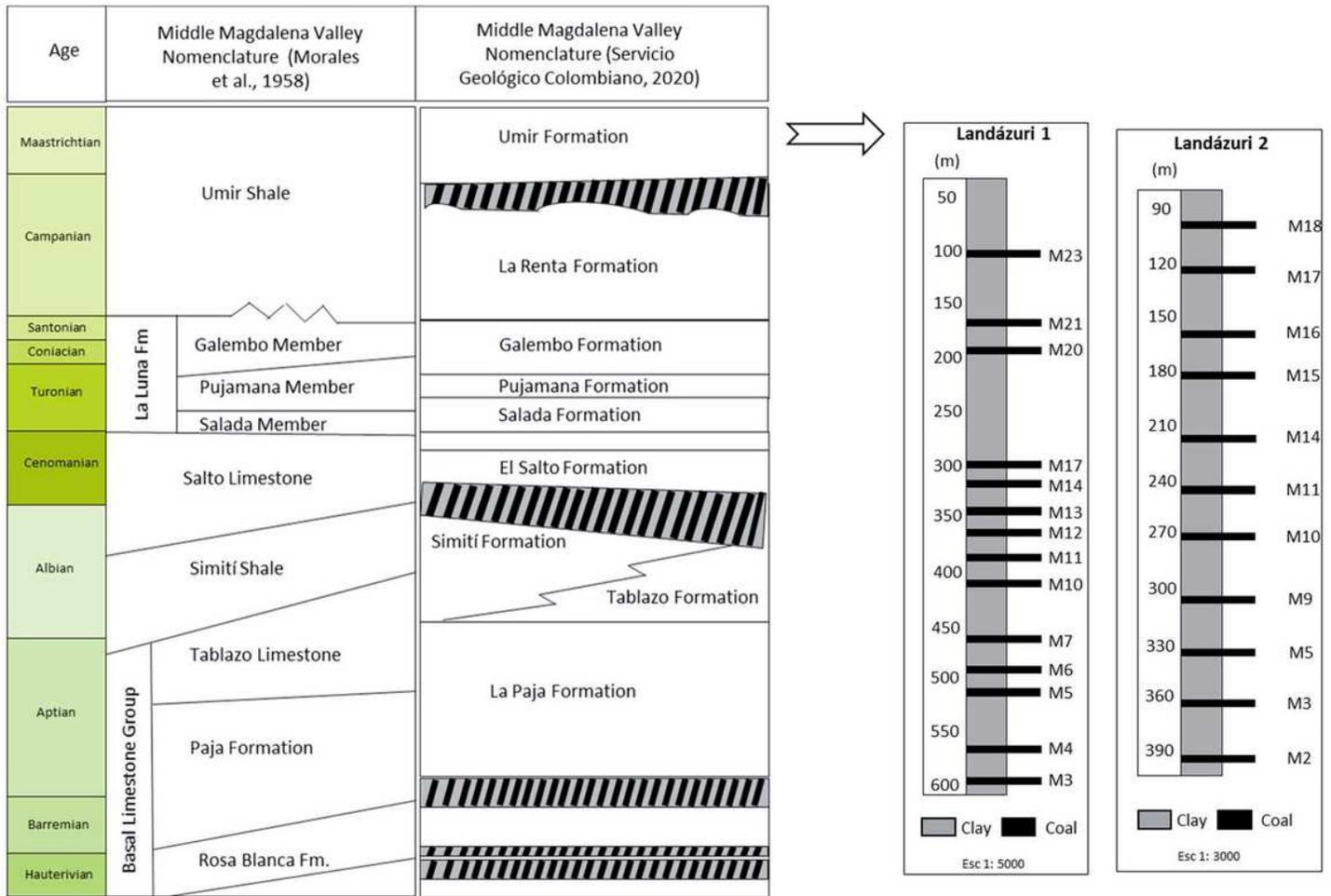


Figure 2

Generalized stratigraphic column of the Upper Cretaceous- Middle Magdalena Valley Basin and Coal seams distribution from Landázuri 1 and 2 wells. Source: (Ortíz et al., 2016; Etayo-Serna, 2019)

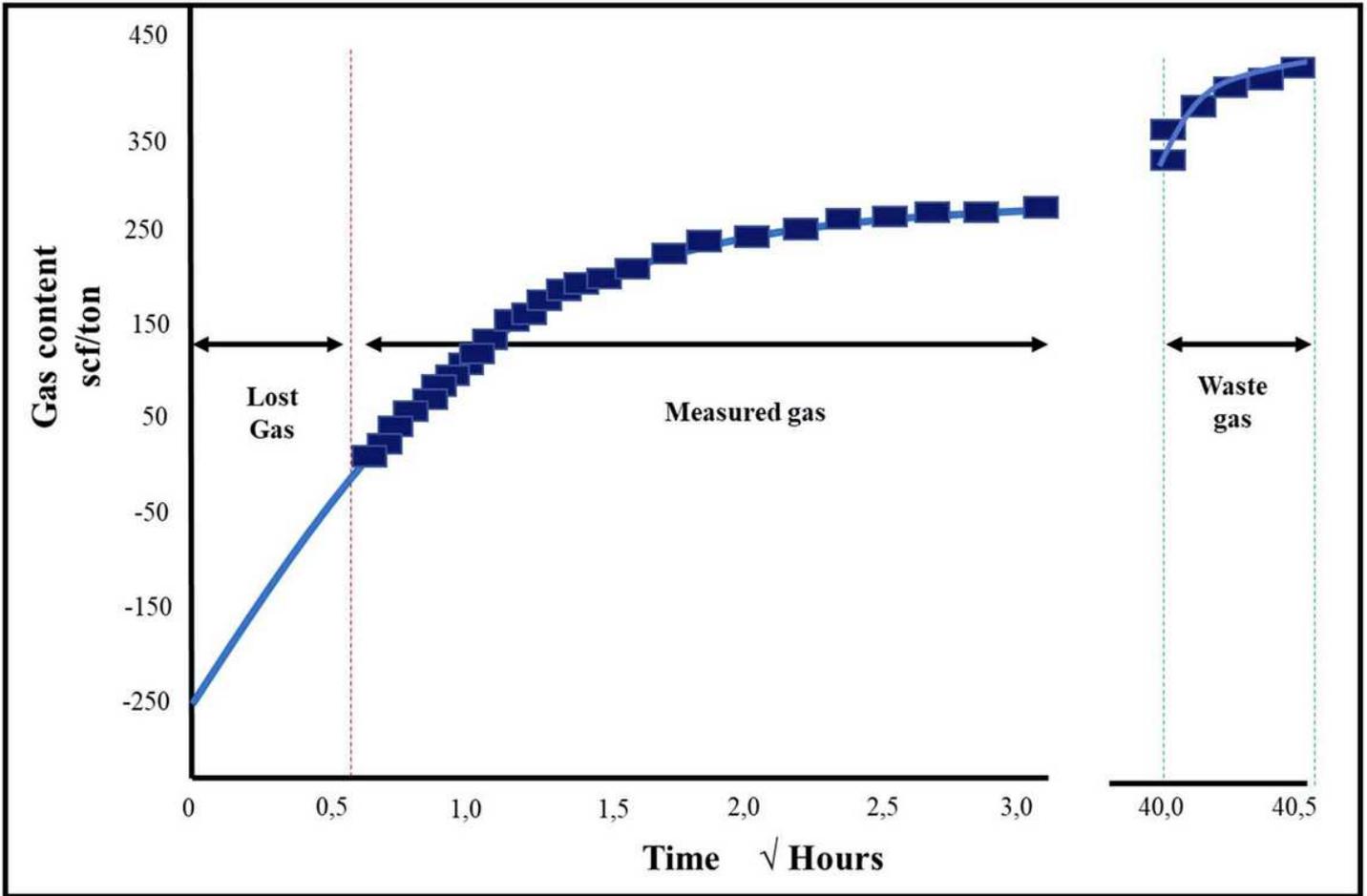


Figure 3

Gas desorption curve (Ortíz et al., 2016)

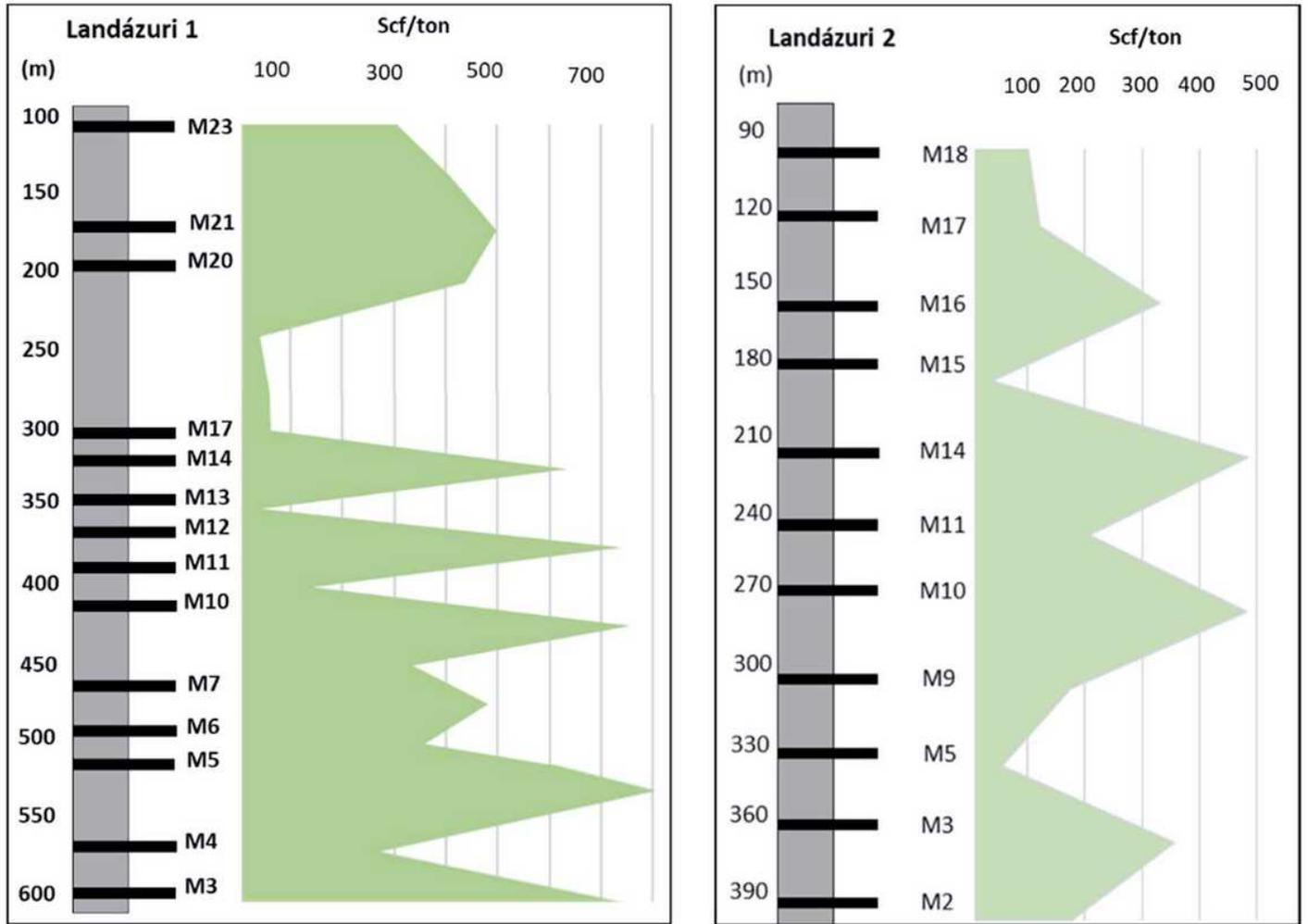


Figure 4

Ash-free gas content (SCF/ton) of coal seams from Landázuri 1 and Landázuri 2 wells (Ortíz et al., 2016)



Figure 5

Coal seams photomicrographs of the Landázuri 1 well after desorption gas process. a. Clay filling inert's empty spaces -M23, b. Framboidal pyrite -M21, c. Devolatilized Colodetrinite -M20, d. Clay mineral matter -M17, e. Fractured Vitrinite and structured Fusinite -M14, f. Inertodetrinite in devolatilized Colodetrinite -M13, g. Framboidal pyrite in Semifusinite -M12, h. Mineral matter -M11, i. Pyrite -M10, j. Vitrodetrinite, k. Colodetrinite -M7, l. Devolatilized Vitrinite -M6, m. Preserved Fusinite and Inertodetrinite- M5, n. Vitrinite and inerts after desorption -M4, o. Degraded inerts - M3



Figure 6

Coal seams photomicrographs of the Landázuri 2 well after desorption gas process. a. Clay minerals - M18, b. Pyrite filling Inertinite's spaces -M17, c. Devolatilized Vitrinite - M16, d. Clay minerals in fractured Vitrinite -M15, e. Colotelinite and Vitrodetrinite -M14, f. Inert in Colodetrinite -M11, g. Semifusinite's spaces filled with mineral matter -M10, h. Empty spaces in Colodetrinite and Semifusinite - M9, i. Inertodetrinite in Colodetrinite -M5, j. Pyrite in Fusinite -M3, k. Fusinite - M2

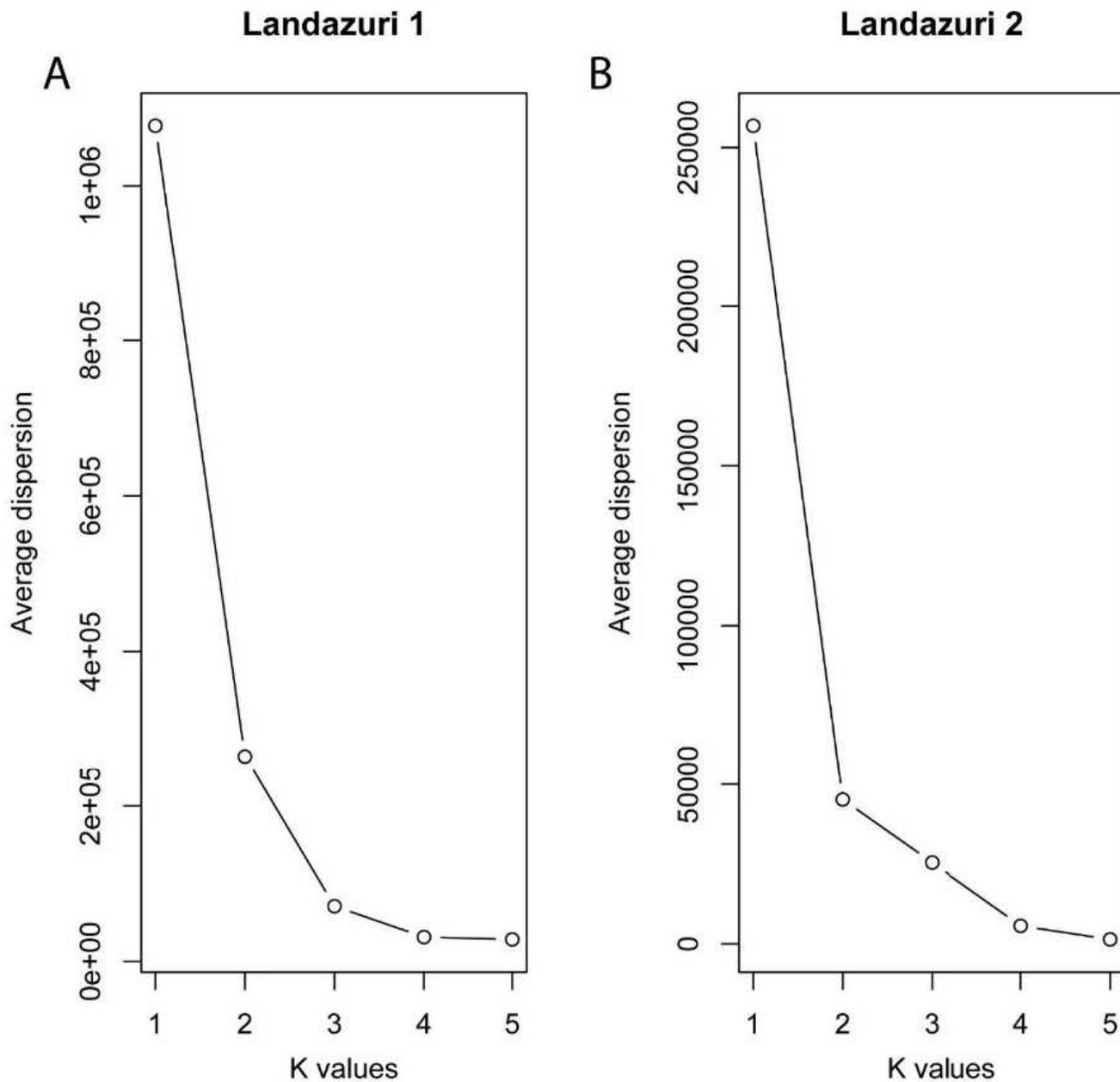


Figure 7

Elbow-plots for (A) Landázuri 1 and (B) Landázuri 2 wells

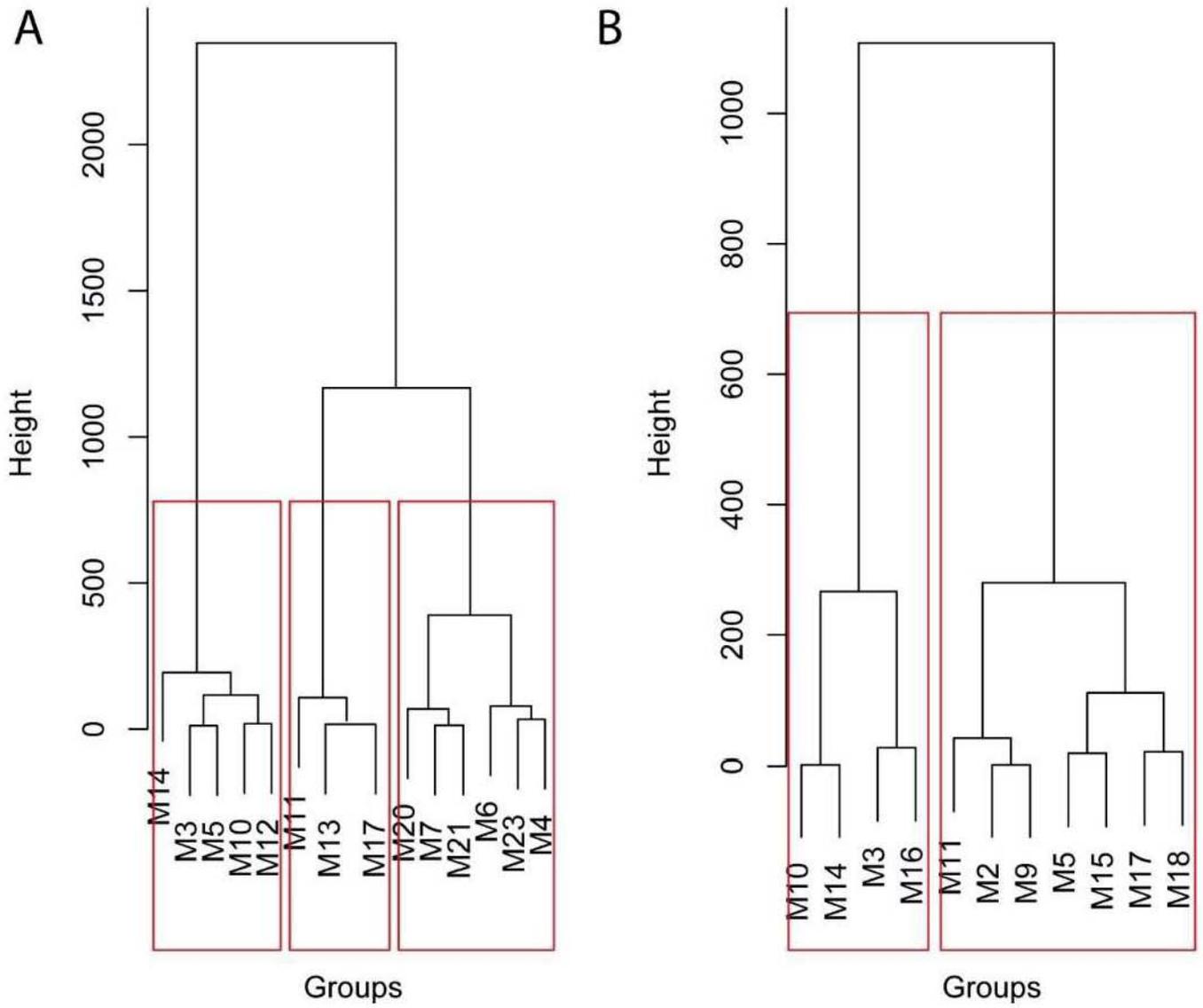


Figure 8

Dendrograms showing results of cluster analysis (A) Landázuri-1 (B) Landázuri-2 wells

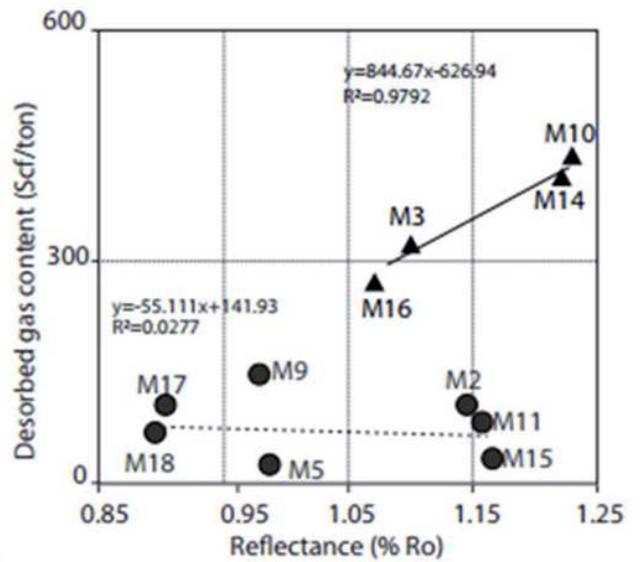
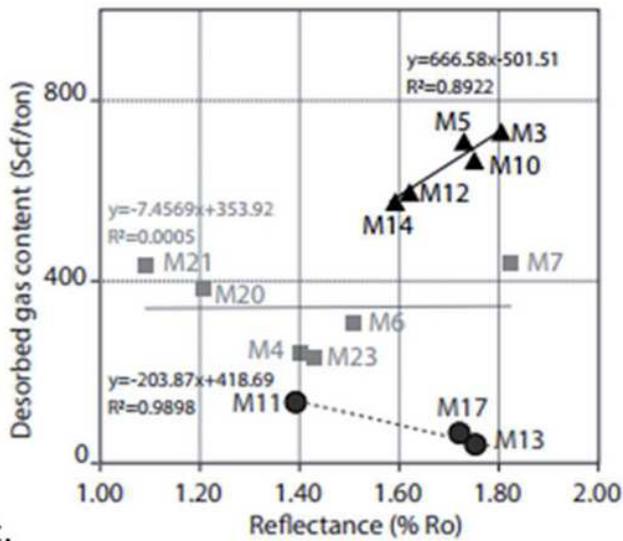
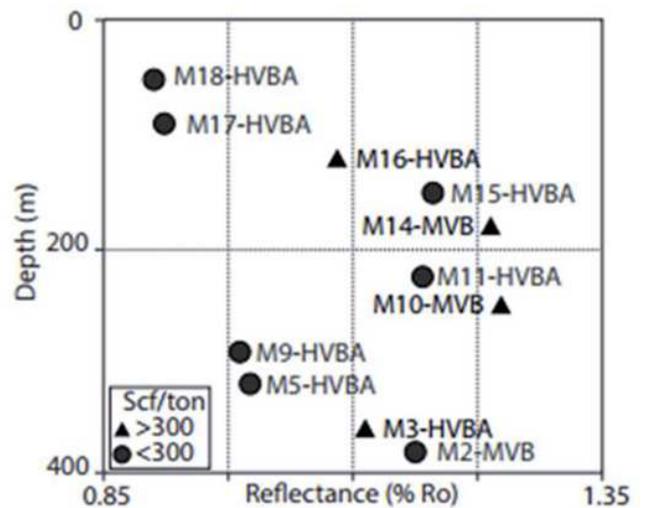
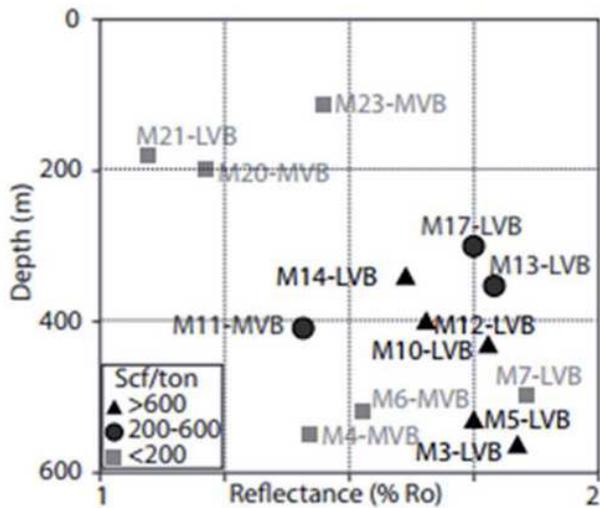
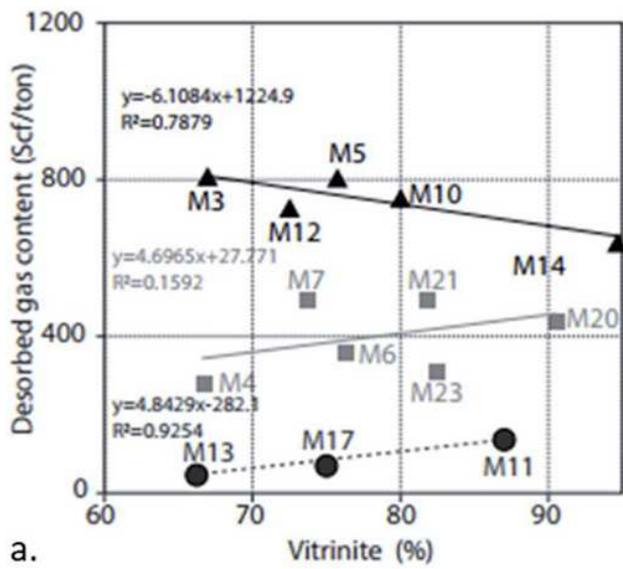
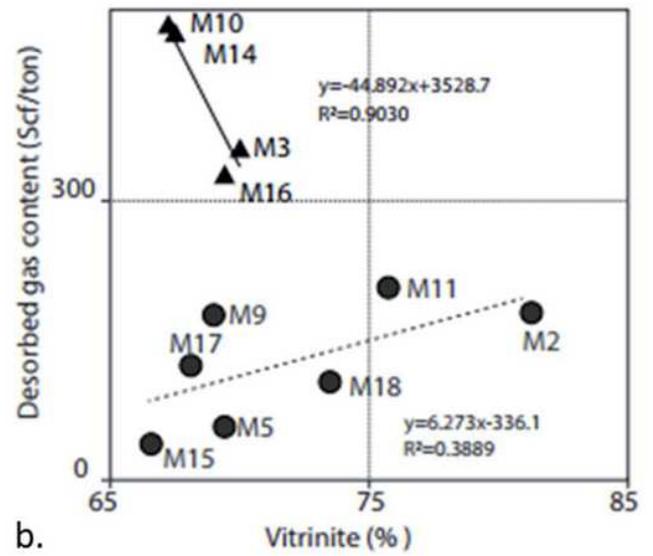


Figure 9

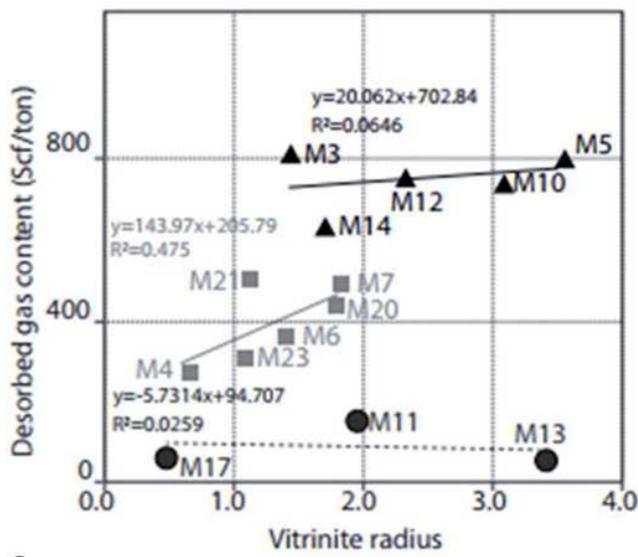
Coal rank (Ro) relation with depth and gas content measured (%Ro) (a, c) Landázuri-1 well. (b,d) Landázuri-2 well. HVBA- High Volatile Bituminous A, MVB- Medium Volatile Bituminous, - LVB Low Volatile Bituminous



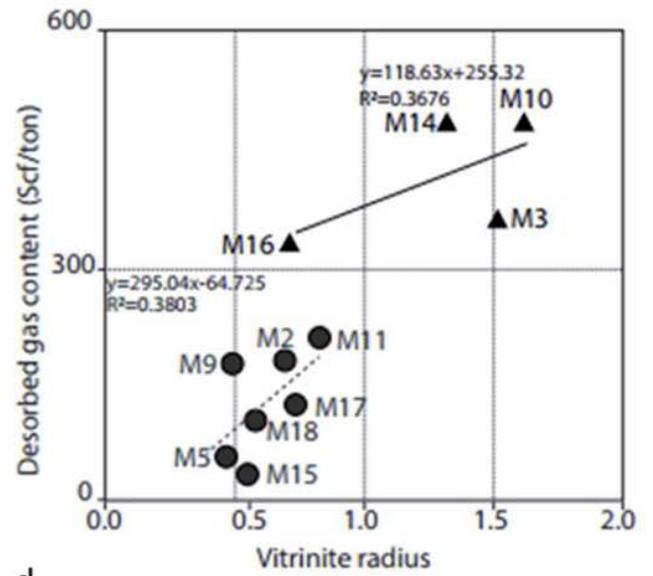
a.



b.



c.



d.

Figure 10

Relationship between Vitrinite percentage (fmmb) and the Vitrinite Radius with the gas content (SCF/ton) (a, c) Landázuri-1 well, (b,d) Landázuri-2 well

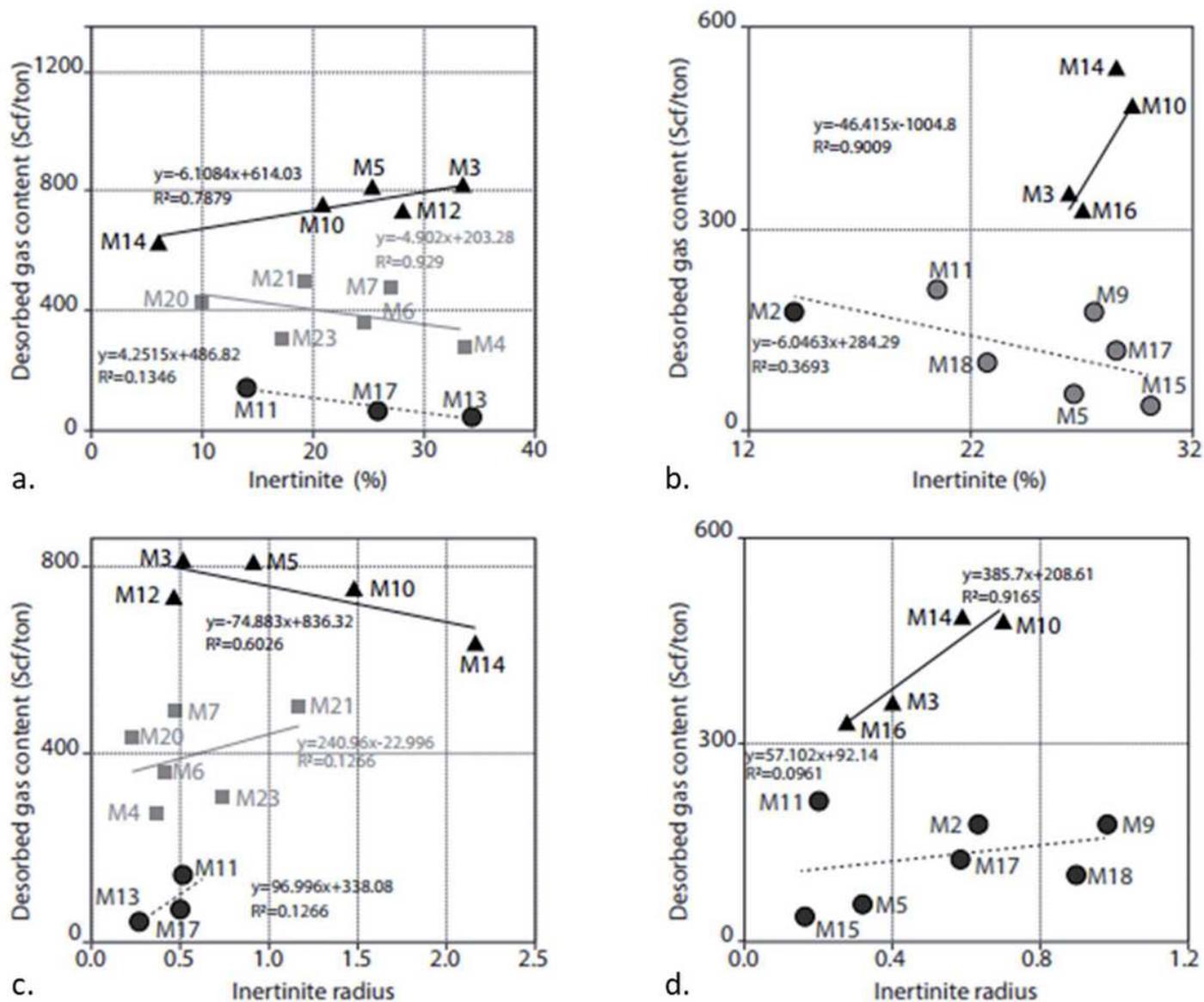


Figure 11

Relationship between Inertinite content (fmmb) and the Inertinite Radius with the gas content (scf/ton) (a, c) Landázuri-1 well, (b, d) Landázuri-2 well

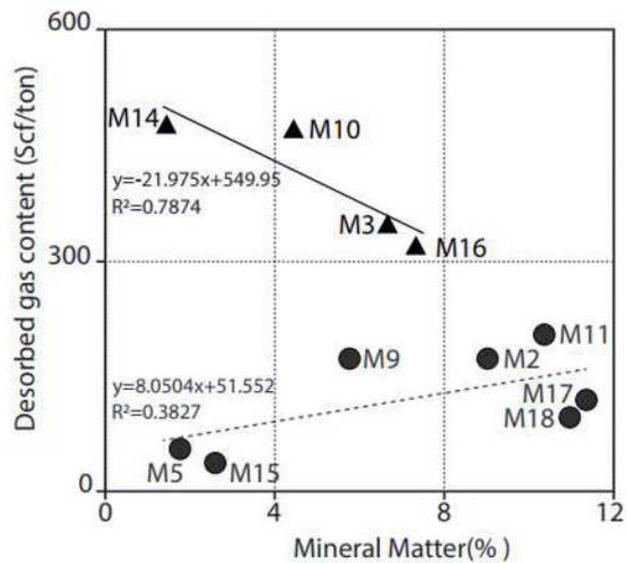
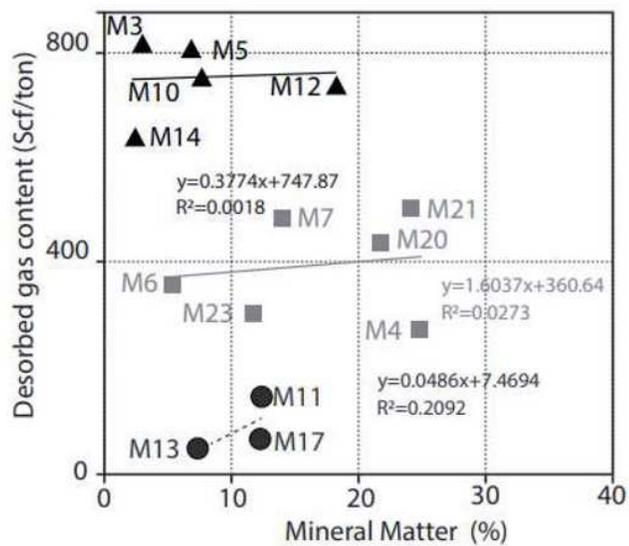


Figure 12

Relationship between mineral matter and the gas content (SCF/ton) (A). Landázuri 1 (B) Landázuri 2

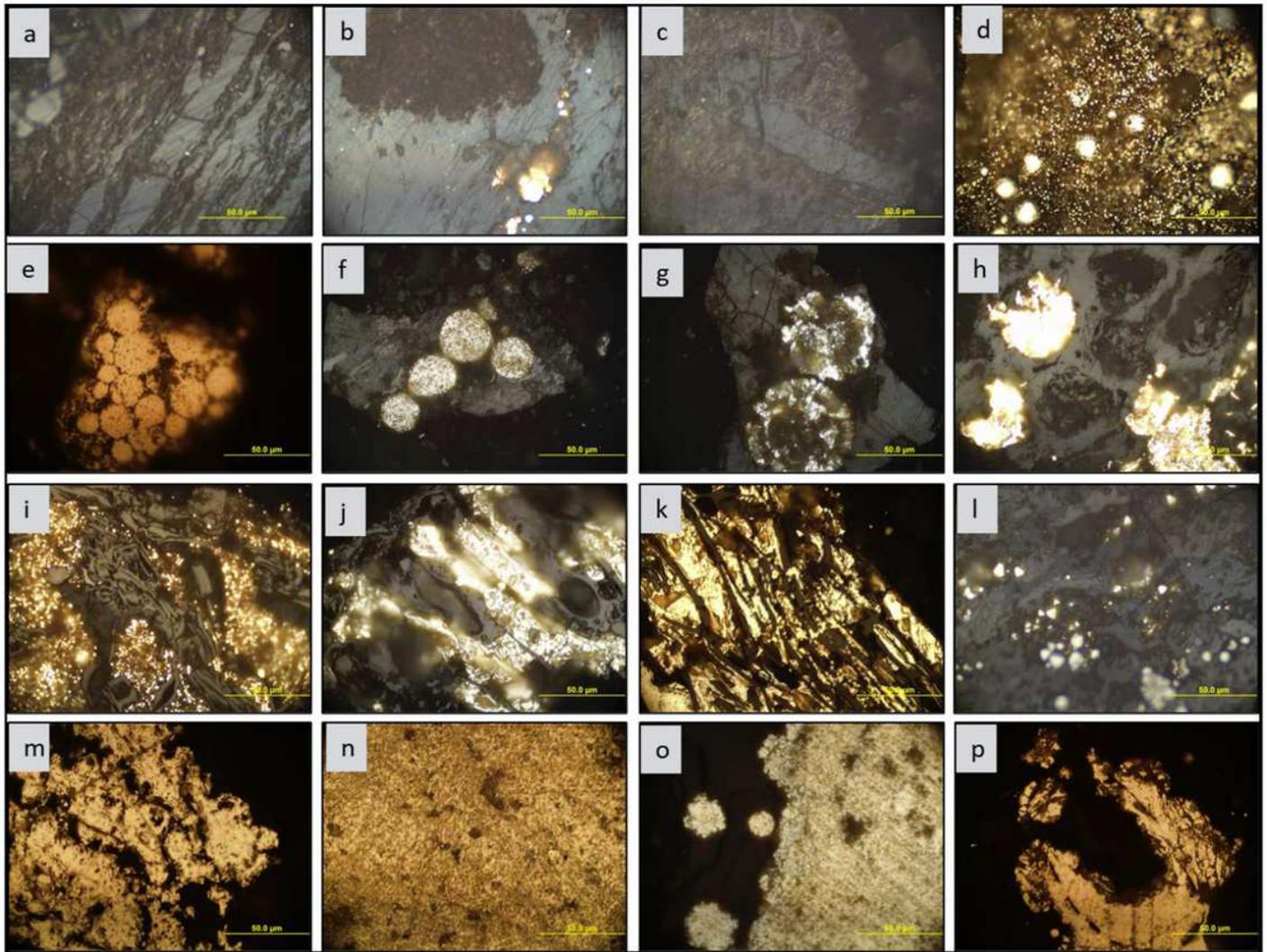


Figure 13

Microphotographs a. Clay minerals in M5- Landázuri-1, b. Clay minerals and pyrite -M17- Landázuri-2, c. Clay and others -M21- Landázuri-1, d. Framboidal pyrite -M17- Landázuri-2, e. Framboidal pyrite -M17- Landázuri-2, f. Framboidal pyrite -M7-Landázuri-1, g. Framboidal pyrite -M7-Landázuri-2, h. Syngenetic-diagenetic pyrite -M3-Landázuri-2, i. Epigenetic pyrite -M17- Landázuri-2, j. Epigenetic pyrite -M2- Landázuri-2, k. Epigenetic pyrite -M17-Landázuri-2, l. Syngenetic framboidal pyrite -M16-Landázuri-2, m. Diagenetic pyrite -M17-Landázuri-1, n. Diagenetic pyrite -M4- Landázuri-1, o. Diagenetic pyrite -M10- Landázuri-1, p. Late diagenetic pyrite? -M17-Landázuri-2.

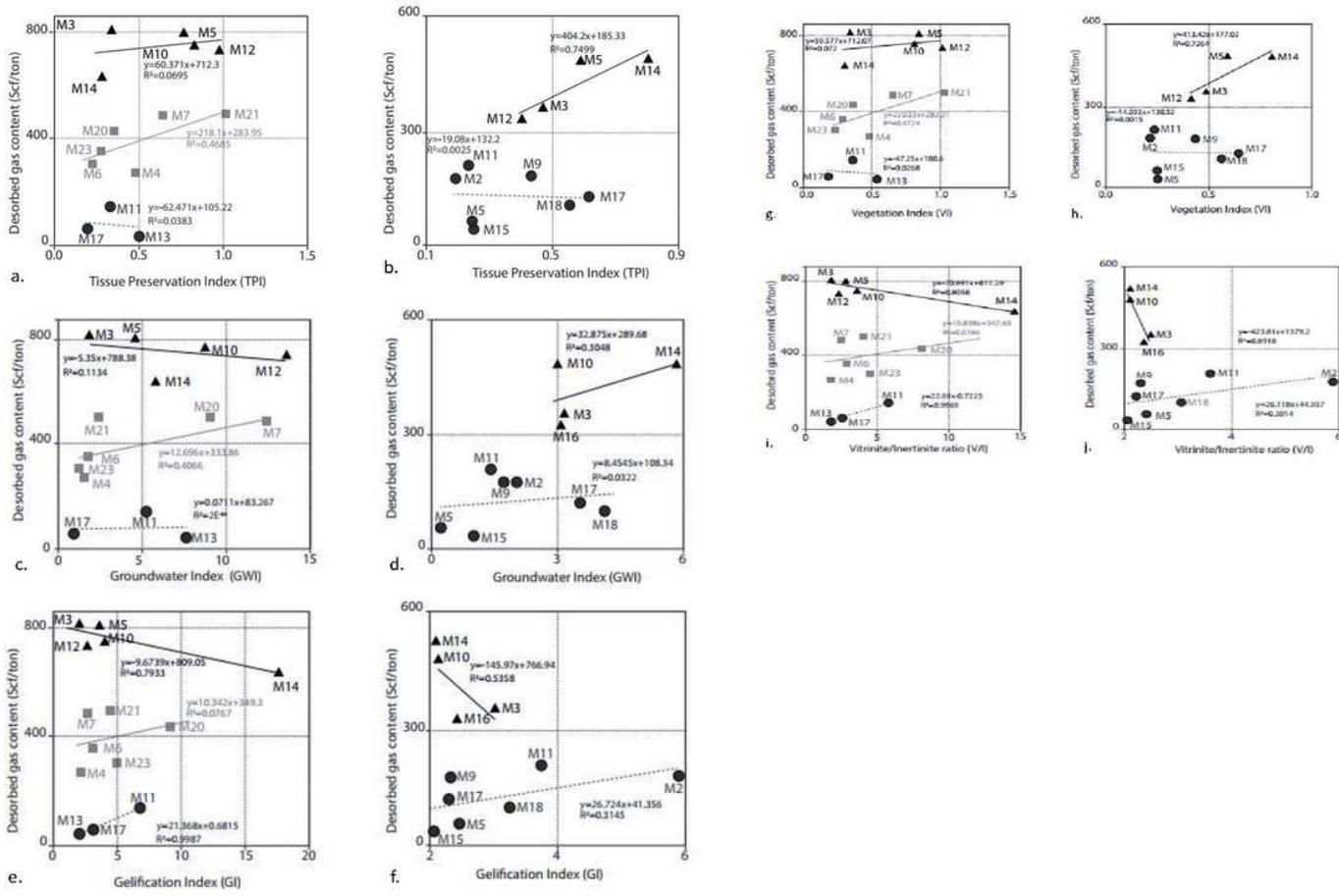


Figure 14

Relationships between gas content (SCF/ton) and petrographic indexes (TPI, GWI, GI, VI, and V/I). Relationships between gas content (SCF/ton) and petrographic indexes (TPI, GWI, GI, VI, and V/I)

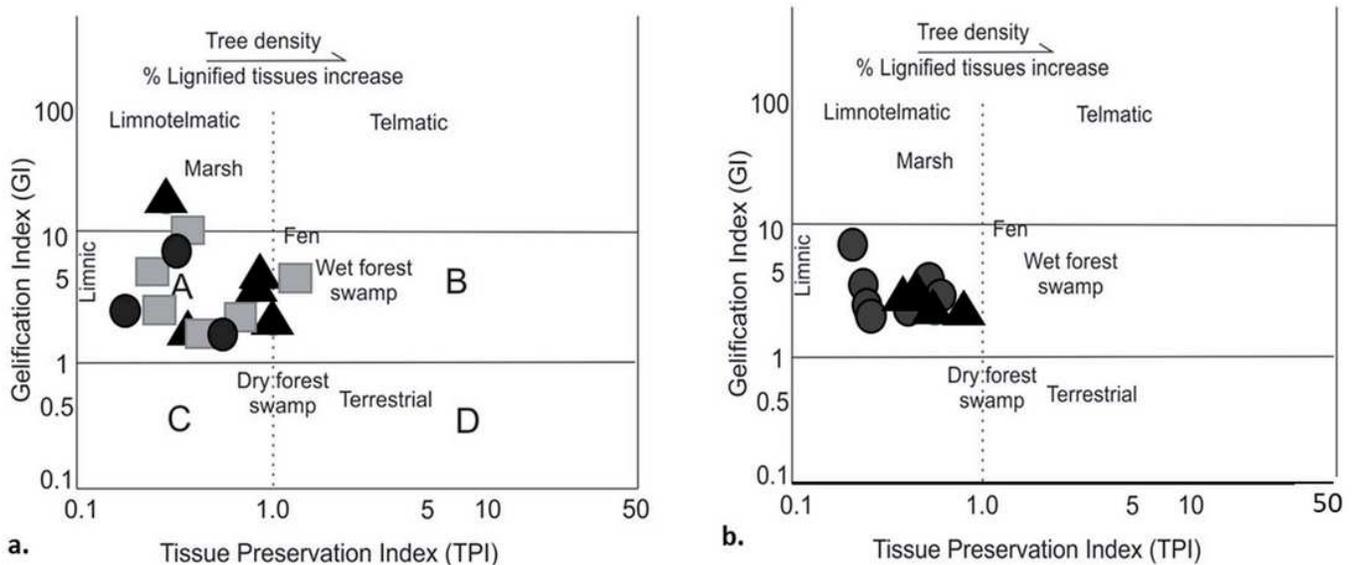
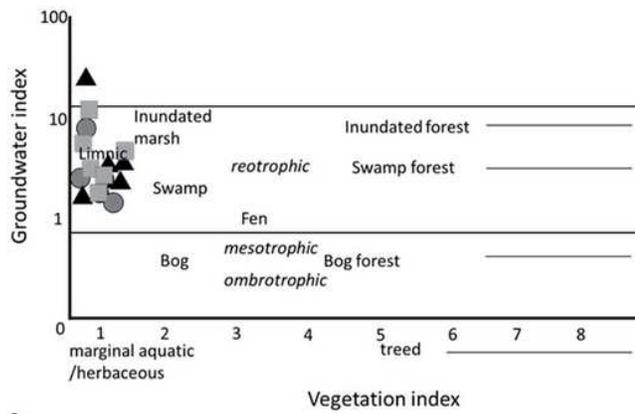
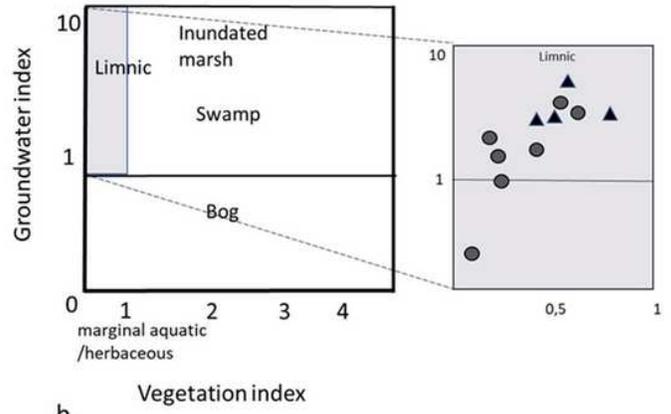


Figure 15

Coal facies (GI, TPI) Source: Diessel (1986) and modified by Kalkreuth et al. 1991. a. Landázuri 1. b. Landázuri 2 A. Vitrinite> Inertinite, Degraded Vitrinite> Structured Vitrinite B. Vitrinite> Structured Inertinite, Vitrinite> Degraded Vitrinite C. Inertinite> Vitrinite Inertodetrinite> Semifusinite- Fusinite D. Inertinite> Vitrinite Semifusinite+Fusinite> Inertodetrinite



a.



b.

Figure 16

Coal facies (GWI, VI). a. Landázuri 1 b. Landázuri 2 (Modified from Calder et al., 1991)

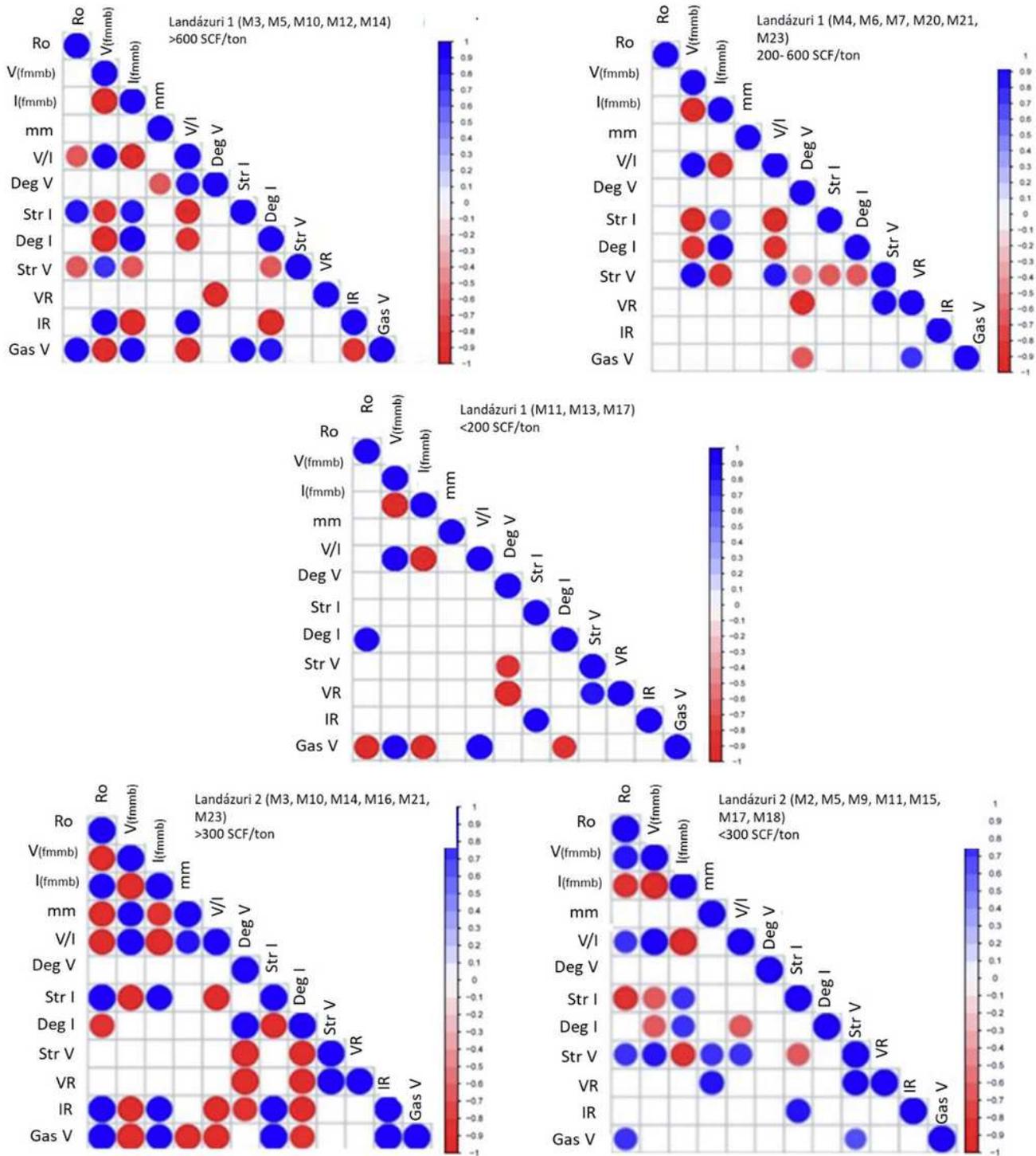


Figure 17

Pearson correlograms showing relationships between pairs of variables for Landázuri 1 and Landázuri-2
 Ro- Average Vitrinite Reflectance, V fmmb - Vitrinite free mineral matter, I fmmb- Inertinite free mineral matter, mm- mineral matter, V/I- Vitrinite/Inertinite radius, Deg V- Degraded Vitrinite, Str I - Structured Inertinite, Deg I - Degraded Inertinite, Str V- Structured Vitrinite, RV- Vitrinite Radius, IR- Inertinite Radius, Gas V- Total gas volume measured SCF/ton

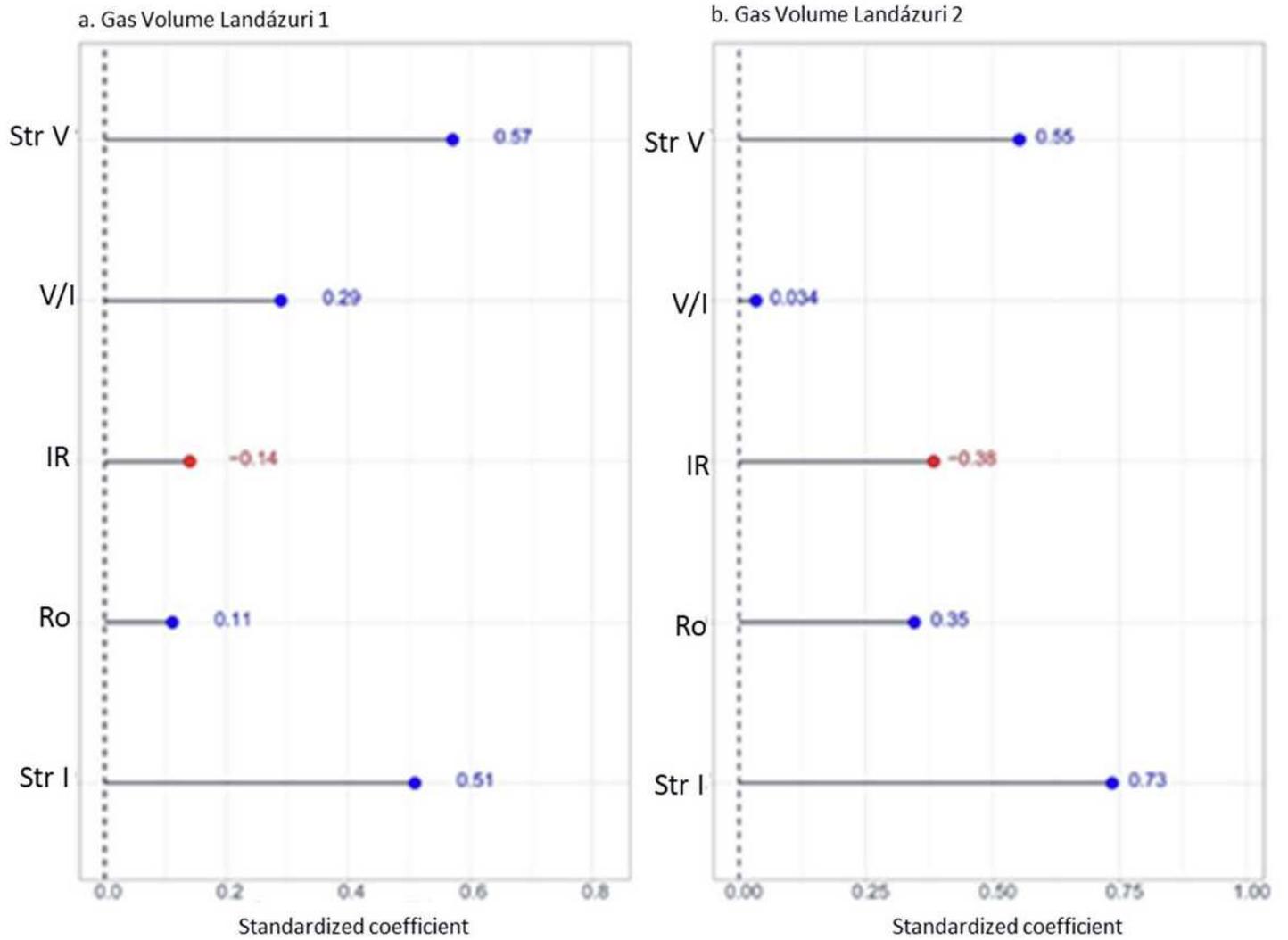


Figure 18

Behavior analysis of predictor variables (Str V, V/I, IR, Ro, Str I) versus response variable (Gas V) for the wells: a. Landázuri 1 and b. Landázuri 2

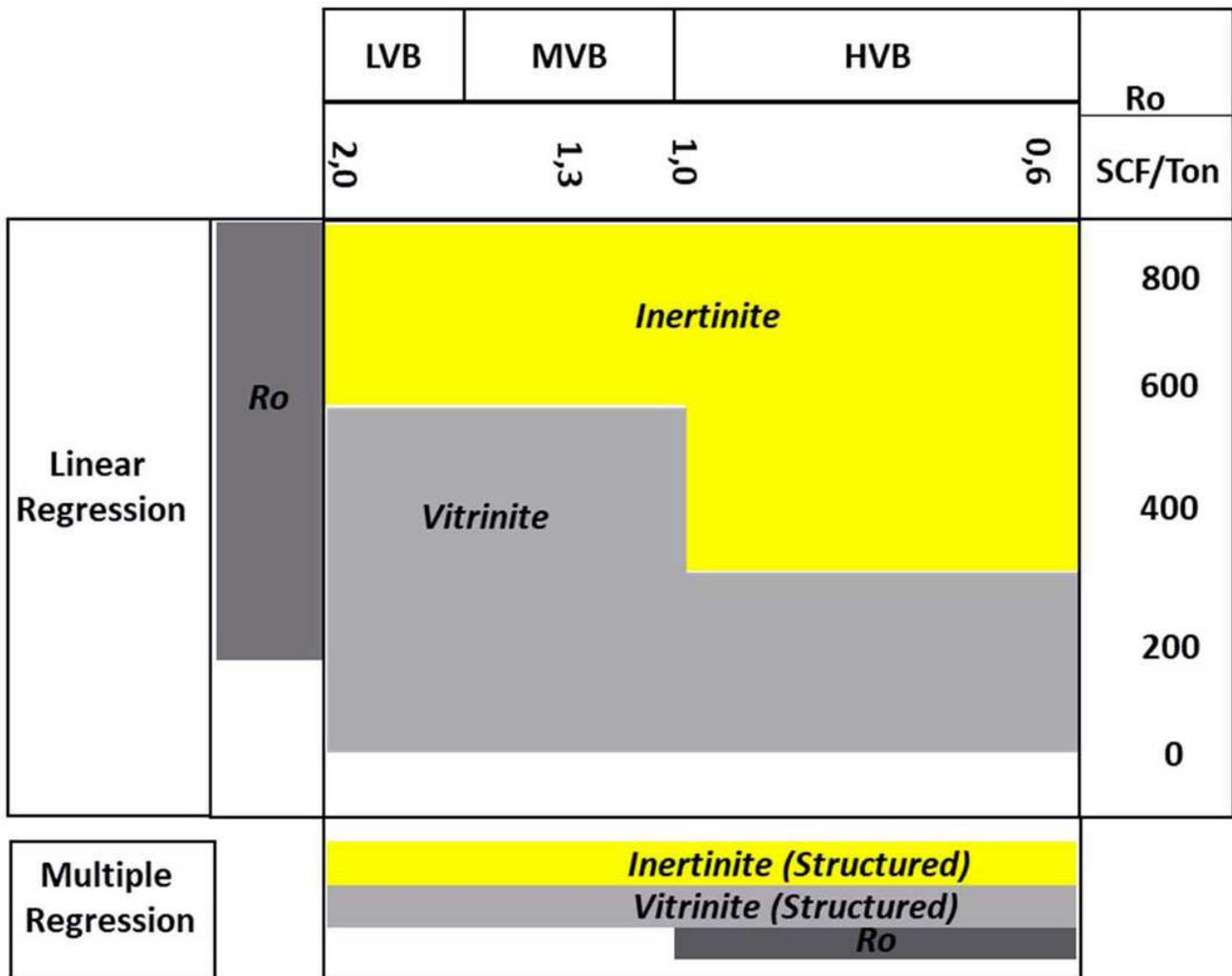


Figure 19

Synthesis of the importance of maceral groups and reflectance for the measured gas content

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