Gómez-Frutos, D., and Castro, A., 2023, **Mafic microgranular enclaves (MMEs) trace the origin of post-collisional magmas:** Geology, https://doi.org/10.1130/G51248.1.

Supplemental Material

Supplemental Text: Analytical Techniques

Supplemental Text: Extended Discussion

Supplemental Data Sheet: Rock Analyses

Supplemental Figure 1: MME Photographs

Supplemental Figure 2: Harker Diagrams
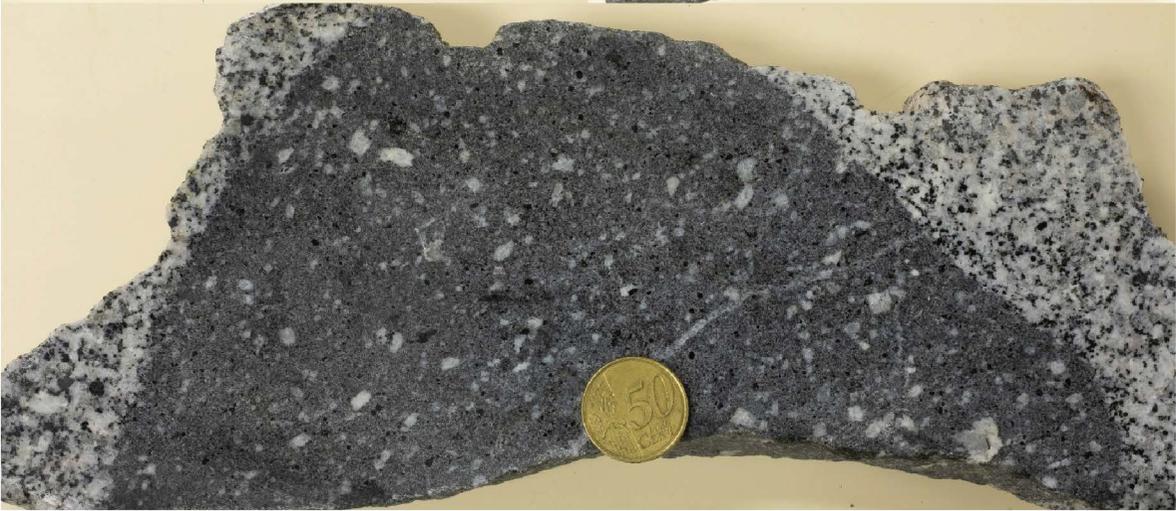
Sampling of mafic microgranular enclaves in the Los Pedroches batholith was made using classic techniques (hammer). All samples were collected in quarries where the land cuts were clean and fresh, devoid of any alteration.

Major and trace elements of the new samples were determined by XRF and ICP-MS at the Centro de Instrumentación Científica of the University of Granada (CIC, UGR). The standard procedures are described in (Baedecker, 1987). Analytical precision is better than 1% for major elements and better than ±5% for 100 ppm Zr. Trace elements, except Zr, were analysed according to the method described by (Bea, 1996). Precision is close to 2% and 5% on concentrations of 50 and 5 ppm, respectively. Trace elements, including REE were analysed on a AGILENT 7900 ICP-MS at the Center for Research in Sustainable Chemistry (CIQSO), University of Huelva, Spain, following digestion in a HF + HNO_3 (8:3) solution, drying and re-dissolution in HNO_3 and later HCl. The ICP-MS analyses were controlled by repeated analysis of the international rock standards AGV-1 and SARM, obtaining a precision better than 10%. Detection limits and analytical uncertainties for the analysed elements are less than 0.1 ppm.

For Sr and Nd isotope determinations, representative samples from each group within Los Pedroches granodiorite suite were digested in a clean room using ultraclean reagents. Samples were selected with the intention to cover all compositional range and check for isotopic equilibrium. They were then analysed by thermal ionization mass spectrometry (TIMS) in a Finnigan Mat 262 spectrometer (University of Granada) after chromatographic separation with ion-exchange resins. Normalization values were $^{86}Sr/^{88}Sr$ = 0.1194 and $^{146}Nd/^{144}Nd$ = 0.7219. Isotopic ratios of $^{87}Rb/^{86}Sr$ and $^{147}Sm/^{144}Nd$ were directly determined by ICP-MS (Montero and Bea, 1998) with a precision, estimated by analysing 10 replicates of the standard WS-E, better than 1.2% and 0.9% (2σ), respectively.

References:

Baedecker, P.A., 1987, Methods for geological analysis. US Geological Survey Bulletin. U.S Geological Survey Bulletin 1770.

Bea, F., 1996, Residence of REE, Y, Th and U in Granites and Crustal Protoliths; Implications for the Chemistry of Crustal Melts. Journal of Petrology, v. 37, p. 521–552.

Montero, P., & Bea, F. 1998, Accurate determination of $^{87}Rb/^{86}Sr$ and $^{147}Sm/^{144}Nd$ ratios by inductively-coupled-plasma mass spectrometry in isotope geoscience: an alternative to isotope dilution analysis. Analytica Chimica Acta, v. 358, p. 227-233.

Harker diagrams showing major element oxides (wt%), Mg#, and Intensity (n = 2557) versus SiO₂ (wt%). Symbols: MME 1 (pink squares), MME 2 (dark blue squares), Granodiorites (green circles), Andean-type series (red dashed line).
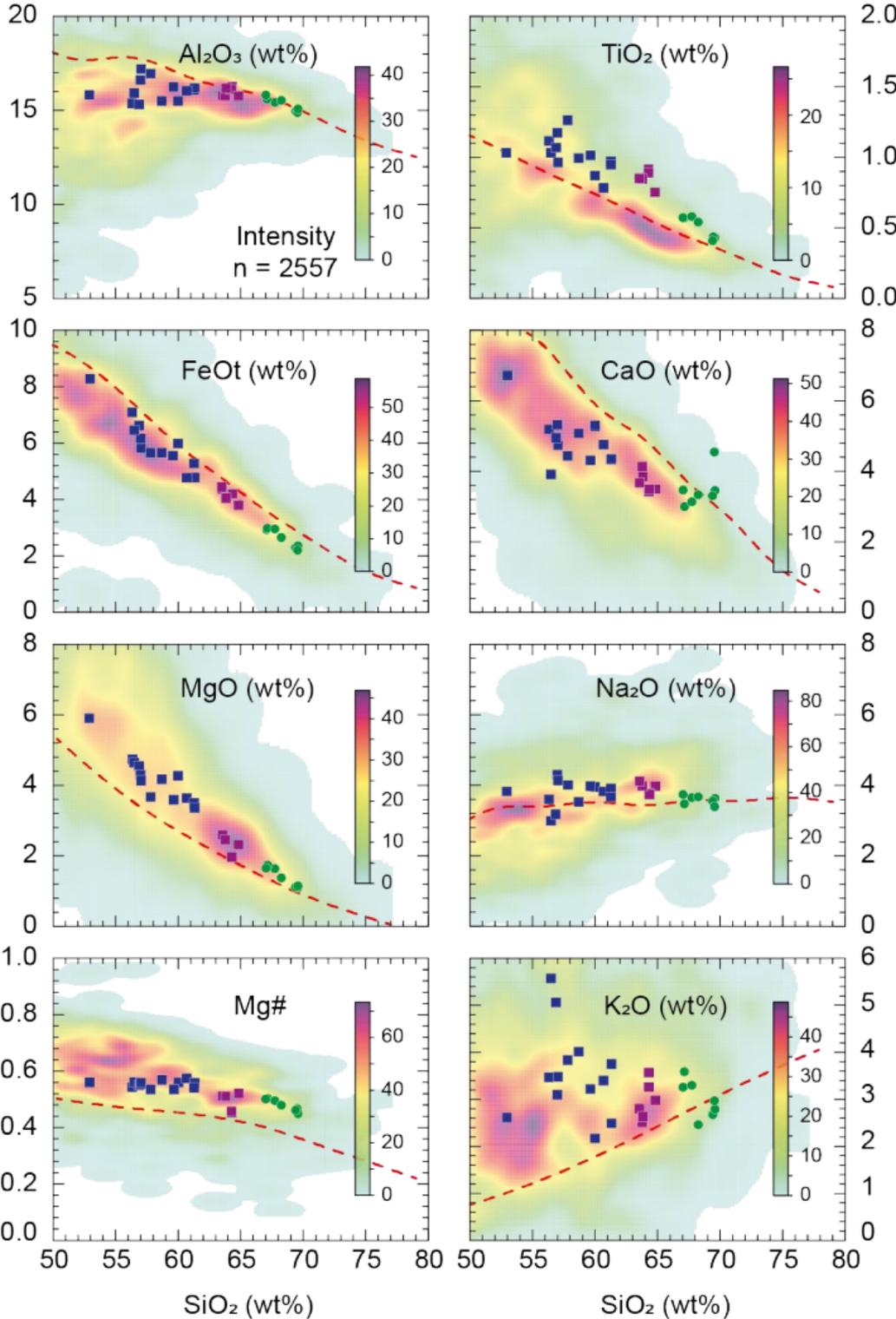
In general, all studied rocks display a continuous trend that has been related to a magmatic fractionation process. Resetting of mineral assemblages to the low-pressure conditions of final emplacement and cooling, makes difficult to know the place of magma differentiation at either the lower crust or the shallow level of emplacement and cooling. Mayor-element trends also are unable to unravel if fractionation occurred in a single stage or by several repeated stages. However, trace element modelling points to a two-stage process, possibly involving different levels of the crust. Because Sr and Y behave distinctly as incompatible ($^{Solid/Liquid}D_i = [i]^{Solid}/[i]^{Liquid} < 1$; being [i] the concentration of element i) or refractory ($^{Solid/Liquid}D_i > 1$), depending on the phase involved, the Sr/Y ratio may be used to monitor fractionation is systems involving Pl, Amp and Px as potential fractionating solids. As the solid/liquid partitioning coefficient ($^{Solid/Liquid}D$) of Sr in Pl is >1 ($^{Pl/Liquid}D_{Sr} > 1$), Sr will tend to be depleted in the fractionated liquid and Y will tend to be enriched as $^{Pl/Liquid}D_Y < 1$. The opposite effect is found if Amp or Cpx are fractionated as $^{Amp/Liquid}D_{Sr} < 1$ and $^{Cpx/Liquid}D_{Sr} < 1$. Both elements, Sr and Y, are incompatible for Opx in andesitic systems ($^{Opx/Liquid}D_{Sr} \approx 0.1$ and $^{Opx/Liquid}D_Y \approx 0.5$; D values from the GERM Partition Coefficients Database, https://kdd.earthref.org/KdD). According to the phenocryst mineral assemblage of porphyritic enclaves, and assuming that Amp aggregates and euhedral Amp crystals were in origin Opx and Cpx phenocrysts respectively, a cumulate composed of variable proportions of Cpx, Opx, Amp and Pl may produce complex variations in the Sr/Y ratios. Figure 10 show the trend of the Pedroches rocks plotted in a Sr/Y versus Y diagram. Fractionated liquids are calculated by batch equilibrium crystallization using the mass balance equation: $C_L = {}^0C_L / [{}^{Solid/Liquid}D + F*(1 - {}^{Solid/Liquid}D)]$; being ^0C_L the initial concentration of Sr or Y in the parental magma; C_L the concentration of Sr or Y in the fractionated liquid at the fraction of liquid (F); and $^{solid/Liquid}D$ the partitioning coefficient. A model of liquids is calculated using the enclave 5Q92, the same used in laboratory experiments (Gómez-Frutos and Castro, 2022; Gómez-Frutos et al., 2023), as the parental magma. A good fit is found using a fractionating assemblage formed by Cpx, Opx, and Pl in the reasonable proportions of 0.6, 0.2, 0.2. Amphibole is not included in this first stage as the transformation of Px into Amp occurred at near solidus conditions, according to thermobarometry estimates, after the accumulation of Px. The proportions of Pl and Cpx are critical as they have a strong weight on the $^{Solid/Liquid}D$ for Sr and Y respectively. Interestingly, whatever the proportions of Cpx and Pl,

there is a maximum value of the Sr/Y ratio in the most fractionated liquid that is lower than the values of granodiorites, the autoliths MME1 and subvolcanic microtonalites. A second fractionation stage, using any of the early fractionated liquids, fairly reproduce the trend of microtonalites, autoliths MME1 and the massive Pedroches granodiorites. In this second episode, Amp can be a dominant phase in the fractionating assemblage, with $^{Amp/Liquid}D_Y \approx 7$ as reported for silicic systems (Sisson, 1994). Accessory minerals as zircon and allanite may have influence in Y fractionation as they have $^{Solid/Liquid}D_Y$ values of about 80 (GERM Partition Coefficients Database, https://kdd.earthref.org/KdD). A proportion of 1% zircon+allanite is considered in the second fractionation curve. Although the Sr/Y ratio is increased by addition of 1% of those Y-rich phases, the attitude of the modelled trend of liquids is not affected, the most fractionated liquids fitting to the compositions of granodiorites. This two-step process is an oversimplification of a process that may have occurred in several stages at different depths within the continental crust. Importantly, enclaves (MM1 and MM2), representing varied stages of the process, were dragged from the walls of ascent conduits carrying important information to the place of observation. Thus, this study opens new perspectives on the petrology of MME as indicators of magma fractionation at variable depths in the generation of post-collisional batholiths.