氧化条件对样品有机碳同位素测定的影响因素讨论

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摘 要 有机碳同位素在全球变化、古气候和古环境恢复研究中的应用日益广泛和深入。有机碳同位素分析的基本原理是在高温下过量的氧气中将样品有机物氧化为 CO_2,通过分离纯化得到纯净的 CO_2 气体送入质谱测定其 $\delta^{13}C$ 值。

在已发表文章中,对于有机碳同位素的研究,人们所采用的氧化温度和氧化时间都有很大差异,但很少系统地研究有机碳同位素分析氧化条件对样品有机碳同位素测定结果的影响。为了解不同的氧化条件是否会造成样品的有机碳同位素分馏,以及不同氧化条件对样品有机碳同位素测定结果的影响程度,笔者采用封管法,选取了不同类型的样品(有机碳标准、植物、表土、黄土以及红粘土等),采用不同的氧化温度:500 ℃、550 ℃、650 ℃、750 ℃、850 ℃、900 ℃以及 950 ℃等,恒温 2.5 h,然后炉冷至室温。

实验结果表明:氧化温度为 850 ℃,恒温时间为 2.5 h,对于有机碳标准、植物、表土和较年轻的黄土样品,足以确保样品有机质氧化充分,$\delta^{13}C$ 值达到稳定,不会产生同位素分馏。

但对于深层黄土和红粘土样品,氧化温度为 850 ℃,恒温时间为 2.5 h,其 $\delta^{13}C$ 值不能达到一个稳定值,仍有偏正趋势。这可能是由于深层样品成岩化作用强,样品中含有一些含碳的矿物包裹体,随温度上升到一定程度,才逐渐分解,释放出气体。对于深层黄土和红粘土样品,采用 850 ℃甚至更高的氧化温度其 $\delta^{13}C$ 值仍未达到稳定,合适的氧化温度需进一步的研究来确定。

关键词 碳同位素 土壤有机质 氧化条件 条件实验

A Discussion on the Effects of Oxidation Conditions on $\delta^{13}C$ of Organic Matter

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Abstract The carbon isotopic composition of organic matter in soil has been widely used in global change research and in reconstruction of paleovegetation and paleoclimate. The basic principle of organic carbon isotopic analysis lies in transforming the organic matter into CO_2 under the superfluous oxygen condition at high temperature, and then separating and purifying the resultants so as to get pure CO_2 for determining their $\delta^{13}C$.

In the study of organic carbon isotope composition, various oxidation temperatures and oxidation durations have been used, but researches about possible effects of oxidation conditions on $\delta^{13}C$ of organic matter are very insufficient. In order to understand whether various oxidation conditions may result in organic carbon fractionation, and what the degree of possible effects of various oxidation conditions on $\delta^{13}C$ of organic matter is, the authors selected various types of samples (standard material for organic carbon, plants, modern soil, loess, red clay, etc.) and used the tube-sealed method to oxidize samples at various oxidation temperatures: 500 ℃, 550 ℃, 650 ℃, 750 ℃, 850 ℃, 900 ℃ and 950 ℃. The oxidation temperature was then kept for 2.5 hours, and after that the muffle was cooled to room temperature with door closed.

According to the experimental results, the oxidation temperature of 850 ℃ and the constant temperature time of 2.5 hours are enough to assure adequate oxidation of organic matter for such samples as standard material for organic carbon, plants, modern soil and younger loess, with no fluctuation of their $\delta^{13}C$ values and no fractionation.

Nevertheless, for older loess and red clay samples at oxidation temperature of 850 ℃ and constant temperature time of 2.5

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hours, the $\delta^{13}C$ values cannot reach constancy and still have a somewhat positive trend. This is probably attributed to strong diagenesis of older samples which might contain some carbonaceous mineral inclusions. With the increase of oxidation temperature, these carbonaceous mineral inclusions begin to break down and release CO_2 gas. For these samples, 850 ℃ or even higher oxidation temperature cannot guarantee the stableness of $\delta^{13}C$ values of the organic matter, and hence further experiments are necessary for determining appropriate oxidation conditions.

Key words carbon isotopes soil organic matter oxida tion condition conditional experiments