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三种干燥方式对粪污厌氧残余物化学性质的影响

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摘要:为揭示干燥方式对粪污厌氧残余物化学性质的影响,开展了真空冷冻干燥、风干干燥和热风干燥对粪污pH值、氨氮、总氮、含盐量、总有机碳和溶解性有机碳的影响,对客观认识粪污理化性质和粪污厌氧残余物干燥方式的优选提供依据。研究结果显示真空冷冻干燥处理方式各化学指标相比于风干干燥和热风干燥方式数值较大,表明真空冷冻干燥处理过程能够维持粪污厌氧残余物化学形态和较高的含量。在此基础上,比较发现真空预冻3 h和预冻6 h对各化学指标的影响差异不显著。建议真空冷冻干燥并且预冻3 h作为粪污厌氧残余物相对较适宜的干燥方式,不仅能够较少地破坏粪污化学性质,同时具有操作简便、节省时间等优点。

关键词:粪污厌氧残余物; 真空冷冻干燥; 风干干燥; 热风干燥; 化学性质

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The effects of three kinds of drying methods on the chemical properties of fecal anaerobic residues

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Abstract: This study was used to reveal the effects of different drying methods on the chemical properties of fecal anaerobic residues, which provided the theoretical basis for choosing suitable drying measures. After fecal anaerobic residues were dried by vacuum freeze-drying, air-drying, and hot air-drying, a series of evaluation indices was tested, which included pH value, ammonia nitrogen, total nitrogen, total salinity, total organic carbon(TOC), and dissolved organic carbon(DOC). The results showed that vacuum freeze-drying had the least loss of chemical components of the three methods, which indicated that the vacuum freeze-drying process could maintain high physical and chemical properties. In this study, the effects of different pre-freezing times on the chemical properties of fecal anaerobic residue were also analyzed. There was no significant difference between 3 hours of freeze-drying and 6 hours of freeze-drying in the characterizations of the fecal anaerobic residues. Thus, freeze-drying for 3 hours is suggested as the best pretreatment because of its easy operation and short time.

Keywords: fecal anaerobic residue; vacuum freeze-drying; air-drying; hot air-drying; chemical property

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粪污厌氧残余物是沼气发酵后剩余的半固体物质,含有丰富的有机质、腐植酸、氨基酸、氮、磷、钾和微量元素,还田施用能够提高土壤肥力、改善土壤结构,增强土壤持续生产能力^[1]。干燥是粪污厌氧残余物最主要的初级处理方式,粪污厌氧残余物干燥有利于原料的保存、深度处理和进一步的科学的研究。而不同干燥方式对粪污厌氧残余物的理化性质影响巨大,选择适宜的干燥方式对于优化资源化处理方案和科学技术研究具有一定的意义。

传统的干燥方式如风干或日晒方式能耗低、设备投入少,但干燥时间长,产品性质变化较大^[2]。近年较多采用的方式是热风干燥或热泵干燥,以及热风或热泵辅助其他方式干燥,这种方式干燥周期短,但能耗高、设备投入多。最近新兴起的真空冷冻干燥技术被广泛地应用到生物、医药和食品领域。Oddone 等^[3]研究表明真空冷冻干燥促进大冰晶的形成,加速了冰的升华并减缓了解吸过程。Dong 等^[4]发现真空冷冻干燥能够很好地保存咖啡豆有机酸和单不饱和脂肪酸。

目前粪污厌氧残余物常用干燥方式有风干干燥、热风干燥、微波冻干和真空冷冻干燥等^[5],综合比较不同干燥方式对粪污厌氧残余物理化性质的研究还较少,而真空冷冻干燥方式对粪污厌氧残余物干燥效果也还未见报道。本文拟通过比较真空冷冻干燥、风干干燥和热风干燥等方式对粪污厌氧残余物理化性质差异,并在此基础上优化真空冷冻干燥预冻时长,从而提出粪污厌氧残余物最佳干燥方式,为更加准确地揭示粪污化学形态与含量,以及为科学的研究开展和干燥方式的优选提供理论依据。

1 材料与方法

1.1 试验材料

粪污厌氧残余物采自天津宁河种猪场规模化养殖能源环境中全混式沼气发酵工程(CSTR),该工程有效反应容积 500 m³,以猪粪和水为原料,日消耗粪便 10 t, 粪污厌氧残余物产出量为 2.05 t·d⁻¹,该沼气发酵工程长期稳定运行。粪污厌氧残余物基本理化性质为含水量 88.84%,pH 值 8.84,铵态氮(NH₄⁺-N)31 mg·g⁻¹,全氮(TN)41.93 mg·g⁻¹,溶解性有机碳(DOC)7.43 g·kg⁻¹,总有机碳(TOC)507.68 g·kg⁻¹,含盐量 0.05%。

1.2 仪器与设备

FD 系列冷真空冷冻干燥机;2XZ-4 型旋片式真空泵;DGG-9240B 型电热恒温鼓风干燥机;DELTA

320 pH 计;DDSJ-308A 电导率仪;KDY-9810 凯氏定氮仪;VarioTOC 测定仪。

1.3 试验设计

1.3.1 试验处理

1.3.1.1 不同干燥方式对粪污厌氧残余物理化性质的影响

真空冷冻干燥(Vacuum Freeze Drying, VFD): 真空冷冻干燥机干燥。称取 500 g 新鲜均匀粪污厌氧残余物均匀覆盖在物料盘中,厚度不超过 10 mm, 放入冷阱预冻 6 h 之后, 将冷冻的粪污厌氧残余物移入真空冷冻干燥室, 腔内压强 3~4 Pa, 低温-80 ℃, 连续干燥 24 h 至恒重。

风干干燥(Air Drying, AD): 自然风干干燥。称取 500 g 新鲜均匀粪污厌氧残余物均匀平铺在物料盘中, 放置在空旷遮光的板房内, 平均室温 8 ℃, 连续干燥一周至恒重。

热风干燥(Heat Air Drying, HAD): 电热干燥箱干燥。称取 500 g 新鲜均匀粪污厌氧残余物均匀平铺在铁盘中, 热风温度 80 ℃, 连续干燥 8 h 至恒重。

3 种不同干燥处理均设置 3 次重复。

1.3.1.2 VFD 处理预冻时长对粪污厌氧残余物化学性质的影响

为进一步优化和探究 VFD 预冻时间对样品各指标的影响,根据真空冷冻干燥设备推荐参数设置了预冻 3 h(VFD3)和预冻 6 h(VFD6)。

VFD3: 称取 500 g 新鲜均匀粪污厌氧残余物均匀覆盖在物料盘中,厚度不超过 10 mm, 放入冷阱预冻 3 h 之后, 将冷冻的粪污厌氧残余物移入真空冷冻干燥室, 腔内压强 3~4 Pa, 低温-80 ℃, 连续干燥 24 h 至恒重。

VFD6: 同上,但放入冷阱内预冻 6 h。

两种预冻时长干燥处理均设置 3 次重复。

1.3.2 测定方法

含水率测定应用恒温箱热风干燥法^[6-7]。pH 值用(水:样=1:1)pH 计直接测定。铵态氮的测定采用凯氏定氮法^[8]。全氮在样品消煮后采用凯氏定氮法测定^[9]。盐度应用电导率仪测定^[10-12]。DOC 采用 TOC 分析仪测定^[13]。总有机碳选用重铬酸钾容量法-稀释热法测定^[14]。

1.4 数据分析

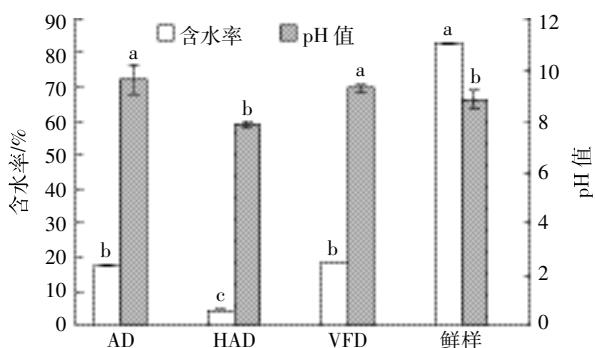
用 Excel 2010 处理数据并作图, 采用 SPSS 软件进行方差分析, 应用 Duncan 方法分析各处理间数据在 $\alpha=0.05$ 水平的差异显著性。

2 结果与分析

2.1 干燥方式对粪污厌氧残余物物理性质的影响

2.1.1 粪污厌氧残余物含水率和pH值变化特征

不同干燥方式下粪污厌氧残余物含水率和pH值见图1。3种干燥处理水分干燥效果总体呈现VFD、AD>HAD趋势,VFD处理样品含水率能够达到与AD处理接近的水平,处理间差异不显著,VFD处理样品含水率比鲜样降低了64个百分点;HAD处理样品的含水率最小,与各处理相比均达到显著差异水平,比AD和VFD处理约低了13~14个百分点。不同处理粪污厌氧残余物pH值呈现AD、VFD>鲜样>HAD趋势。AD处理pH值与VFD处理间差异不显著,分别比鲜样pH值有所升高,维持了鲜样较高的pH值特征。HAD处理pH值比鲜样降低了11%,朝向中性趋势降低,与AD和VFD处理相比差异达到显著水平($P<0.05$)。



不同字母表示处理间差异达0.05显著水平($P<0.05$)。下同

Different letters are significantly different ($P<0.05$). The same below.

图1 不同干燥方式对粪污厌氧残余物含水率和pH值的影响

Figure 1 Characteristics of moisture content and pH value under different drying methods

2.1.2 粪污厌氧残余物TN和NH₄⁺-N含量变化特征

不同干燥方式下粪污厌氧残余物TN和NH₄⁺-N见图2。3种干燥方式粪污厌氧残余物TN含量依次为VFD>AD>HAD。虽然VFD处理TN含量较鲜样降低了30%,但比AD和HAD处理分别高27%和69%,与HAD处理间差异达到显著水平($P<0.05$)。总体来看,3种干燥处理NH₄⁺-N含量趋势为VFD>AD>HAD,处理之间差异分别达到显著水平($P<0.05$),VFD处理NH₄⁺-N含量分别是AD处理和HAD处理的1.5倍和4倍。3种干燥处理NH₄⁺-N含量均比新鲜样品有所损失,VFD处理NH₄⁺-N含量比鲜样降低了35%左右,但处理间差异不显著,HAD处理NH₄⁺-N

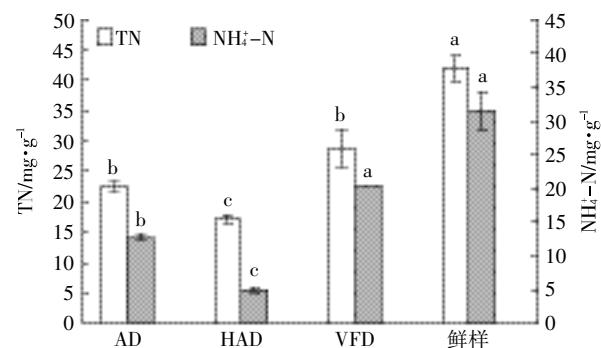


图2 不同干燥方式对粪污厌氧残余物TN和NH₄⁺-N含量的影响

Figure 2 Characteristics of TN and NH₄⁺-N under different drying methods

含量比鲜样降低了84.44%,HAD处理和AD处理与鲜样之间差异达到显著水平($P<0.05$)。

2.1.3 粪污厌氧残余物TOC和DOC含量变化特征

不同干燥方式下粪污厌氧残余物TOC和DOC含量见图3。粪污厌氧残余物TOC含量总体变化趋势为鲜样>HAD、AD、VFD。3种干燥处理TOC含量都较鲜样降低了40%,3个干燥处理之间TOC含量差异并不显著。粪污厌氧残余物DOC含量总体变化趋势为鲜样>VFD>AD>HAD。VFD和AD处理的DOC含量与鲜样间差异不显著,HAD处理DOC含量与其他处理间差异均达到显著水平($P<0.05$)。

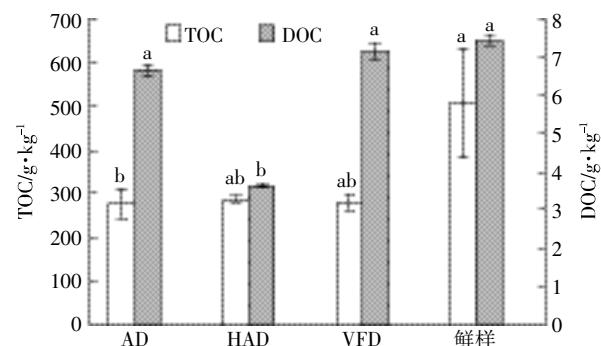


图3 不同干燥方式对粪污厌氧残余物TOC和DOC含量的影响

Figure 3 Characteristics of TOC and DOC under different drying methods

2.1.4 粪污厌氧残余物全盐量变化特征

不同干燥方式下粪污厌氧残余物全盐量见图4。粪污厌氧残余物全盐量总体呈现VFD>HAD>鲜样>AD趋势。VFD处理全盐量比鲜样显著增加,分别是AD、鲜样和HAD的4.7、2.8倍和2.3倍,处理之间差异均达到显著水平。AD、HAD处理与鲜样间差异不显著。

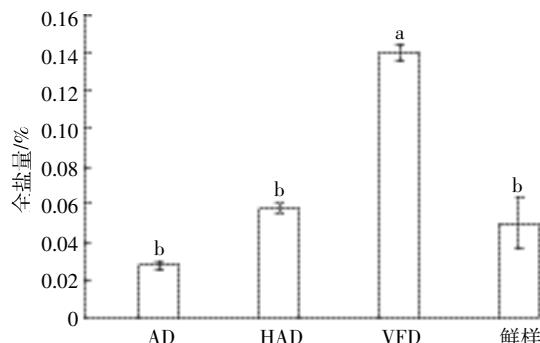


图4 不同干燥方式对粪污厌氧残余物全盐量的影响

Figure 4 Characteristics of total salinity under different drying methods

2.2 VFD 处理预冻时长对粪污厌氧残余物化学性质的影响

预冻时长(预冻3 h和6 h)对粪污厌氧残余物化学性质的影响见表1。两个预冻时长处理含水率、NH₄⁺-N、DOC、TOC、TN、全盐量等指标数值非常相近,所有指标处理之间差异均不显著,真空冻干预冻时长对粪污厌氧残余物理化性质的影响较小。预冻3 h既节约时间与能源,又可达到与预冻6 h相似的干燥效果和稳定的化学性质。

3 讨论

本研究比较了VFD、AD和HAD干燥方式对粪污厌氧残余物干燥效果与化学性质。结果表明VFD和AD干燥所得样品中TN和NH₄⁺-N含量较高,HAD处理含量最低,主要因为高温条件下部分氮素以气体形式分解扩散,而VFD和AD干燥在相对低温条件下有效保持了NH₄⁺-N含量^[15-16]。DOC含量变化特征与NH₄⁺-N相似,VFD和AD干燥处理中DOC含量较高,而HAD处理DOC含量相对较低,主要因为高温显著促进了粪污中易氧化碳和溶解性有机碳的分解^[17],而VFD和AD干燥过程中抗氧化成分和性质得到较好保护^[18],张雨婷等^[19]和高炜等^[20]发现冷冻干燥能够保持铁皮石斛较高的多糖含量,也从侧面给出了相似的研究启示。本研究发现3个干燥处理TOC含量差异

不显著,表明非水溶性有机碳仍然是粪污厌氧残余物中主要形态,并且该部分成分对温度变化不明显。有研究发现与TOC相比,草地土壤DOC的变化更为迅速^[21],并且土壤DOC含量在有机质中所占的比例很小^[22]。VFD处理的粪污厌氧残余物全盐量不仅明显高于AD和HAD处理,甚至高于鲜样。这可能是因为VFD处理存在冻融过程,螯合的离子在冻融过程中被释放,同时有机物矿化率增大,这个推论在土壤的冻融过程中被广泛证实。常宗强等^[23]发现冻融过程对土壤氮矿化有促进作用,罗金明等^[24]研究得出冻融土壤中HCO₃⁻、CO₃²⁻等离子含量显著增加。VFD、AD和HAD处理方式对粪污厌氧残余物理化性质均有较大的影响,但VFD方式与AD和HAD相比影响最小。HAD方式虽干燥时间短,但耗能高,干燥方法工业化应用具有一定局限性,在粪污规模化生产中使用较少^[25-27]。VFD能够实现与AD相近的水分去除效果,本研究也证实VFD处理缩短干燥时间、保持样品化学组成等优势^[28-34],对于了解和掌握粪污本身化学含量和形态具有一定的意义。

VFD干燥前需对样品进行预冻,目的是为了冻结样品中水分以进行真空升华^[35]。预冻时间过长不仅浪费能源和时间,而且会影响样品中化学组成。本研究表明两组预冻时长处理对粪污厌氧残余物化学性质影响差异不显著,均可以获得较好的干燥效果和稳定的化学性质与组成,这与干燥物体的性质和尺寸有关。任红兵^[36]研究发现预冻时间控制在3 h以内即可满足VFD升华过程,与本研究结果高度一致。因而推荐真空冷冻干燥且预冻3 h作为粪污厌氧残余物干燥方式。

4 结论

(1) 真空冷冻干燥、风干干燥和热风干燥对粪污厌氧残余物的pH、NH₄⁺-N、TN、含盐量、TOC和DOC均有较大的影响,但真空冷冻干燥与风干干燥和热风干燥相比影响最小。

(2) 真空冷冻干燥6 h或3 h预冻时长对粪污厌

表1 不同预冻时长对粪污厌氧残余物化学性质的影响

Table 1 Chemical properties of bio-slurry under different pre-freezing periods

处理	pH值	含水率/%	NH ₄ ⁺ -N/mg·g ⁻¹	TN/mg·g ⁻¹	全盐量/%	TOC/g·kg ⁻¹	DOC/g·kg ⁻¹
VFD3	8.92±0.92a	16.01±0.14a	19.8±0.11a	29.53±2.37a	0.141±0.004a	270.81±9.24a	6.72±0.17a
VFD6	9.26±0.10a	18.75±0.14a	20.44±0.08a	28.81±3.04a	0.140±0.005a	279.59±19.52a	7.16±0.22a

注:数值为平均值±标准差,相同列的不同字母表示在0.05水平下差异显著。

Note: The values are mean ± standard deviation, and the different letters of the same column indicate significant difference at 0.05 level.

氧残余物化学性质影响差异不显著。

(3)预冻3 h的真空冷冻干燥处理方式对样品化学性质影响最小,干燥方便快捷、操作简便,推荐作为农业生产和科学使用的研究使用的干燥处理方式。同时在生产上或科研上的应用还应综合考虑成本和研究目标。

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