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富氮低氧+磷化氢防治储粮害虫 技术的实仓应用研究（中英文）

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摘要: 为防治澳大利亚主要仓储害虫, 开发富氮气调技术作为熏蒸剂的辅助手段。然而, 由于成本问题, 一直影响其在行业推广应用。本研究利用中国长顺安达公司的先进膜分离技术, 与传统的制氮技术(变压吸附/PSA)进行性能比较, 在西澳 Kwinana 的 CBH 粮食港, 开展实仓应用实验, 以解决阻碍其推广的运营和成本问题。为满足澳大利亚严格的标准操作条件, 长顺安达专业团队对港口设备进行了升级改造, 目标消杀成本设定为 0.50 澳元/吨粮(磷化氢的高端熏蒸成本)。研究表明, 旧的 PSA 技术的运营成本为 2.43 澳元/吨粮, 该技术在产生和维持控制害虫所需的 99%氮气浓度水平方面也显示出操作局限性。相比之下, 使用膜技术制氮的成本为 0.99 澳元/吨粮, 其他性能增益还包括运行 4.5 天内即可产生所需的氮气浓度, 且可维持 14 天以完全控制害虫。

关键词: 粮食储藏; 储粮害虫管理; 氮气储藏; 变压吸附制氮机; 膜分离制氮机

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Delivery and Adoption of Nitrogen/Low Oxygen and Nitrogen + Phosphine Technology for the Management of Grain Storage Pests in Commercial Silos (Chinese and English versions)

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Abstract: Nitrogen technology has been developed as a complementary technology with fumigants, for control of key pests in Australian grain storages. However, market feedback from industry was its cost which remained as a barrier of uptake. Therefore, a commercial scale trial was set up using advanced membrane technology to assist industry overcome operational and the cost barriers for the uptake of the technology. A new generation membrane technology was sourced from Changshun Anda, a China-based company. The unit was deployed at the CBH grain port of Kwinana, Western Australia to compare performance against an older nitrogen generation technology (Pressure Swing Absorbance/PSA). To meet the strict Australian standards operating conditions the port the unit was upgraded by Changshun Anda specialist team. The target benchmark was set to \$0.50 per tonne of grain (the high-end cost of a phosphine fumigation). Research demonstrated an operational cost for the older PSA technology of \$2.43 per tonne of grain. The technology also displayed operational limitations in its ability to generate and maintain the required level of nitrogen purity necessary to provide insect control (99%). In comparison, the generation of nitrogen using membrane technology cost \$0.99 per tonne of grain. Other performance gains were generation of required nitrogen purity within 4.5 days of operation and ability to maintain purity for the required 14 days to provide full control of pests.

Key words: grain storage; stored grain insect management; nitrogen storage; Pressure Swing Adsorption (PSA) nitrogen generators; Membrane Separation (MS) nitrogen generators

尽管磷化氢仍然是控制仓储害虫最常用的气体熏蒸剂,但还需要探索其他选择。气调是一个人们目前感兴趣的领域,其中氧气、二氧化碳和氮气的浓度以及温度和湿度都是可调节的。通过调节谷物、豆类和油菜籽中的气体组分来控制害虫,因为没有氧气的情况下大多数害虫无法长期生存。这项技术在该行业有着悠久的历史^[1-3],但作用过程相当缓慢,需要长达数周的时间,尤其是较低的温度下(低于 15 °C)。

2016 年,澳大利亚粮食行业设立了一个磷化氢替代品开发投资项目,即利用氮气气调技术作为熏蒸剂的可选择手段。该项目以变压吸附(PSA)技术制备 99% 氮气,研究了杀虫基础数据和最佳氮气操作程序。变压吸附(PSA)技术是使用特定的吸附材料(例如沸石、活性炭和分子筛),首选在高压下从空气中分离吸附目标气体组分,然后转到低压以解吸吸附的气体组分。为了从受益和效率的角度解决成本障碍并推动投资机会,在西澳开展了一项农场实验,该实验为相关种植者带来了正面的经济效益,氮气处理过的谷物符合有机

食品分类,从而获得了市场溢价。

然而,根据工业市场的反馈,成本仍然是采用该技术的一个障碍;但对仓房气密性的高要求还是另一障碍,尽管随着害虫对磷化氢的抗药性推动了粮食行业进行仓房气密性升级。最近,采矿业通过利用膜技术在制氮成本方面取得了重要进展,研究人员将该技术引入粮食行业,克服了氮气气调的应用推广障碍。氮气膜通过选择性渗透穿过氮膜壁的原理来分离气体,与 PSA 技术相比,在操作方面具有若干优势。

1 方法与材料

1.1 变压吸附制氮技术商业化实验

变压吸附技术在澳大利亚 CBH 公司运营的几个港口码头现场使用。实验在 Kwinana 港进行,现场的移动式变压吸附发生器以 30 m³/h 的速度产生 99.5% 的纯氮。在西澳大利亚 Kwinana 码头选择的筒仓是混凝土立筒仓,已用薄膜密封,使其气密性达到一般磷化氢熏蒸的要求。使用 PSA 氮气发生器的实验是在一个 550 t 的“星形”筒仓中进行的,大约装有 20 t 大麦。“星形”单元是在建

造过程中在四个“主”圆柱形单元之间形成的空隙。为了提高储藏能力，并为码头提供更多的谷物分仓储存的选择，这些空间密封后也可以用于储存粮食。

实验所用筒仓的谷物出口槽安装了球阀和 3 英寸凸锁接头，以方便充氮处理。筒仓还安装了内部管道，以便在正常熏蒸情况下对熏蒸气体进行环流。顶空出口管道将用作筒仓内部大气的净化。顶空氧气浓度也将在此位置进行监测。氧气浓度由 Draeger XAM-7000 和 Toxipro 个人防护监测器组合记录。筒仓顶部空间和环境温度数据由 Onset Hobo 温度/相对湿度监测仪记录。

1.2 膜分离制氮技术商业化实验

默多克大学从中国长顺安达购买的膜分离制氮机可生产 $60 \text{ m}^3/\text{h}$ 纯度为 99.5% 的氮气。在实验开始之前，该系统已按照澳大利亚电气标准进行了升级和认证。这项工作是在长顺安达专家的支持和建议下，由签约的第三方完成的。此外，设备还具有便携性/移动性的特点，便于在不同地点之间以及在大型商业场所（如 Kwinana 港口设施）内进行转移。压缩机和膜组件氮气发生装置都安装在一辆卡车上。卡车配备拖车，可以运输所需的电力（柴油发电机），以备现场无电力供应时使用，或作为现有电力供应的备用电源（图 1）。



图 1 由空气压缩机、膜组件氮气发生装置和柴油发电机组成的移动式熏蒸装置，摄于 Kwinana 码头粮食仓库外。

Fig.1 Mobile fumigation unit comprising compressor, membrane separation nitrogen generator and diesel generator outside grain storage at the Kwinana terminal.

实验在一个 2 300 t 的圆柱形筒仓中进行，仓内装满了大约 2 200 t 大麦。由于该装置的制氮能力较高，因此使用了较大容量的筒仓。膜单元的流速和氮气纯度可以调节，从而提高流速或氮气纯度。

经过短暂的预热期后，平均可生产 $85 \text{ m}^3/\text{h}$ 纯度为 98.5% 的氮气。首先，为了能够快速吹扫净化

料仓，我们选择了提高流速和降低纯度。一旦观察到氧气浓度有了合理的下降，就将流速降至 $40 \text{ m}^3/\text{h}$ ，以将氮气纯度提高到 99.5%。膜实验的设置和应用程序与 PSA 实验相同。从筒仓顶部进行内部气体组分吹扫净化和监测。照片中的管道连接到筒仓的顶部空间。这是氧气浓度监测的位置。在关闭膜发生器之前，关闭混凝土层的“闸”阀，以便在料仓中形成轻微的正压。

如图 1 所示，Toxipro 氧气监测仪悬挂在黄色安全锥的顶部。这表明，耗尽的氧气环境不会对经过熏蒸区的工作人员造成危险。事实上，CBH 的工作人员只能检测到管道末端 30 cm 范围内的氧气含量低于正常水平。

1.3 膜分离制氮技术实仓实验

与明格诺-欧文集团（MIG）合作，在西澳明格诺粮食区进行了实仓实验。选择用于示范的两个筒仓是现代化的 75 t 高锥底筒仓，工厂安装了地面磷化氢应用、热虹吸系统和泄压阀。首先对筒仓进行了压力测试，以确保适合氮气处理。利用鼓风机通过泄压阀向筒仓施加压力，观察连接的数字手持式压力计达到 250 Pa 时关闭风机并开始计时，记录压力衰减到设定值一半（125 Pa）时所用的时间，这就是仓房的压力半衰期。两个筒仓的“半衰期”均大于 5 min，证实了其适用性。

其中一个筒仓（筒仓 1）装的是 2016 年的小麦，装载量为 80%。另一个筒仓（筒仓 2）装有当年的新小麦，装载量为 100%。这些粮食均没有经过磷化氢处理。在筒仓 1 中观察到高浓度的二氧化碳，这表明小麦中可能存在害虫。从两个筒仓中扦取谷物样品，以了解其谷物质量特性，并在实验结束时与处理后的谷物样品进行比较。一部分谷物样本也被放置在默多克大学收获后生物安全实验室的恒温恒湿培养设施中，以评估自然感染的谷物昆虫在 5 周内的进一步发展情况。

在筒仓顶部空间和底部锥体两个位置安装了监测管线，以测量筒仓的氧气浓度。在两个筒仓的顶部空间安装了温度记录仪，并在环境中安装了一个温度记录仪，以监测环境条件。为便于向筒仓中充氮，在筒仓热虹吸循环系统中安装了一个分接系统。气密性紫外线稳定球阀用于构建应用装置。氮气通过安装在筒仓底部锥体上的分接

系统进入筒仓，从热虹吸管通向磷化氢熏蒸室。
该系统 and 关键部件如图 2~9 所示。



图 2 正在向 75 t 筒仓充入氮气。该膜分离制氮装置 (60 m³/h, 99% 纯氮) 可完全移动, 由柴油发电机提供动力。
Fig.2 Nitrogen application underway to 75 tonne silos. This Membrane Nitrogen generation unit (60 m³/hour, 99% pure nitrogen) is completely mobile being powered by a diesel generator.



图 3 通过 60 m³/h、99% 纯度的膜分离制氮机生产氮气, 直径为 3/4 英寸的软管连接到出口。
Fig.3 Nitrogen is produced via 60m³/hr, 99% purity Membrane separation Nitrogen Generator. Hose of diameter 3/4" connected to outlet.



图 4 分接系统安装在现有的热虹吸管管道上。热虹吸管由 90 mm 雨水 PVC 管组成, 从筒仓顶部空间通向地面磷化氢应用室。磷化氢室具有进入筒仓底部的入口管道, 以完成再循环回路。
Fig.4 Tap system installed in existing thermosiphon pipework. Thermosiphon pipework consists of 90 mm stormwater PVC pipe leading from headspace of silo to ground level phosphine application chamber. Phosphine chamber has entry duct into base of silo, to complete the recirculation loop.



图 5 在筒仓锥体和侧壁的连接处, 热虹吸管的直径减小到 40 mm 排水排污管 (DNW), 直径变小的管道与地面磷化氢施放室相连, 充氮分接系统就是安装在这条管道上的。

Fig.5 At the join of the cone and sidewall of the silo, the thermosiphon pipe reduces in diameter to 40mm Drain and Waste (DNW) pipe. This reduced diameter pipe connects with the ground level phosphine application chamber. It is this pipe where the Nitrogen application tap system was installed into.



图 6 氮气通过分接头系统进入料仓, 该系统包含紫外线稳定球阀和凸轮锁接头, 便于软管连接。
Fig.6 Nitrogen is applied to the silo via tap system incorporating UV stable ball valves and camlock fittings for ease of hose connection.



图 7 为了便于安装分接头系统, 我们切割并拆除了 550 mm 一段的管道 (40 mm)。考虑到分接头系统的额外重量, 安装了一个支架来支撑管道。使用两个 40 mm 橡胶套管连接 40 mm 管道。在橡胶套管上安装了四个软管夹, 并将其固定, 以完成气密安装。
Fig.7 A length (550 mm) of the 40 mm pipe was cut and removed to facilitate installation of the tap system. A brace was installed to support the pipe given the additional weight of the tap system. Two 40 mm rubber sleeves were used to connect onto the 40 mm pipe. Four hose clamps were installed to the rubber sleeves and fastened to complete the gas tight installation.

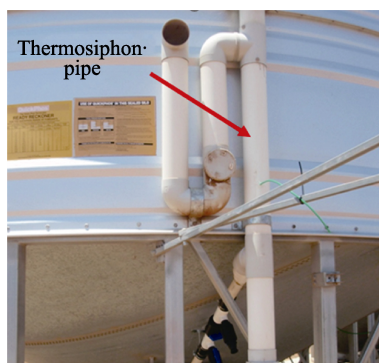


图 8 氮气应用系统安装完毕后,要进行压力测试,以确保筒仓的气密性足以维持足够的氮气浓度。以气密著称并安装了热虹吸系统的筒仓需要泄压阀来补偿温度波动,以防止筒仓损坏。通过向筒仓施加一定体积的空气(250 Pa),并使用手持式压力计和秒表进行压力测试,当压力衰减到一半(125 Pa)时,就是压力半衰期,以分钟为单位。当压力半衰期达到 5 min 时,表明适用于施用氮气。

Fig.8 Once the Nitrogen application system had been installed, a pressure test was conducted to ensure the silo was gas tight enough to maintain adequate concentrations of N_2 . Silos sold as gas tight and fitted with thermosiphon systems require pressure relief valves for compensation of temperature fluctuations to prevent silo damage. Pressure tests are conducted by applying a volume of air (250 Pa) to the silo and with a handheld manometer and stopwatch, timing the decay of that applied pressure to half (125 Pa) giving its 'half loss time'. A value in minutes. The silos for these trials returned a 'half loss time' of 5 minutes indicating suitability for the application of Nitrogen.



图 9 一旦筒仓顶部空间和锥体中的氧气浓度恢复到 1% 或更低的值,表明存在 99% 的氮气,就可以减少氮气产量并关闭排气球阀。短暂地持续低流量(约 $10 \text{ m}^3/\text{h}$) 氮气将在料仓中形成轻微的正压,这将有助于料仓保持高浓度氮气的能力。然后关闭氮气施用球阀并抽出软管。由于氮气流量很小,因此可以在仍有氮气流出的情况下松开软管。然后打开管线球阀,让氮气通过热虹吸系统在筒仓内循环,这取决于每天的温度波动。

Fig.9 Once the Oxygen concentration in the Headspace and cone of the silo return values of 1% or less, indicating the presence of 99% Nitrogen, the Nitrogen production can be reduced and the exhaust ball valve closed. Briefly continuing a low flow (around $10 \text{ m}^3/\text{hour}$) of Nitrogen will build a slight positive pressure in the silo, which will aid in the silos ability to maintain high concentrations of Nitrogen. The N_2 application ball valve is then closed and hose withdrawn. The hose can be unfastened whilst still flowing Nitrogen as the flow is minimal. The inline ball valve is then opened allowing Nitrogen to circulate around the silo via the thermosiphon system, as dictated by the daily temperature fluctuations.

将氮气应用于筒仓时将进料盖关闭锁好。筒仓内部大气通过热虹吸管最靠近筒仓壁的球阀在地面排出。

这种应用系统可将氮气排入筒仓底部。具体做法是关闭在线接头,通过熏蒸室将产生的氮气导入筒仓底部。熏蒸室的出口与筒仓底部相连,完成了热虹吸系统的循环。氮气随后扩散谷物内部,置换出内部空气。充氮时,筒仓盖要锁紧,这样就能通过热虹吸管将置换净化后的空气导出筒仓顶部。然后,通过安装在充氮口附近的另一个球阀旋塞将大气排出地面。

膜分离制氮机设定为输送 $33 \text{ m}^3/\text{h}$ 99.4% 的纯氮。最初设定的流量较高,因为该装置可以输送高达 $60 \text{ m}^3/\text{h}$ 的 99% 氮气,但在这种情况下,较高的流量导致泄压阀迅速起泡。在 $33 \text{ m}^3/\text{h}$ 的情况下,泄压阀轻轻起泡。氮气通过直径为 20 mm 的编织软管输送到料仓,软管上安装有 3/4 英寸凸锁配件。在整个充氮期间,对料仓的顶空和锥体进行监测,以确定可以停止充气的时间。充氮一直持续到顶空和锥体的氧气值恢复到 1%,表明氮气含量达到 99%。

在施用期结束时,首先关闭大气排气球阀,在筒仓内形成轻微的正压。然后关闭充气口,抽出软管。为使热虹吸系统再次正常运行,然后打开中央在线接头,使氮气根据每天的温度波动在筒仓内循环。

2 结果与分析

2.1 制氮技术商业规模实验

主要发现如下:

星仓需要大约 84 h 才能将氧气浓度降至 5% 以下;

又过了 84 h, 氧气浓度才降到 3%;

再监测 3 天,但仓内氧气浓度已稳定在 3.3%, 没有观察到进一步的减少;

总应用时间约为 168 h。

PSA 制氮机是主电源装置,因此很难获得能耗数据来计算处理成本。不过,根据 Lake Grace 农场主使用相同型号和容量的 PSA 制氮机,柴油发生器每小时吹扫净化消耗约 5 L 柴油,平均每

升柴油价格为 1.45 澳元，处理的总运行成本约为 1 218 澳元或 2.43 澳元/吨粮。

2.2 膜分离制氮技术商业规模实验

由于这是膜分离制氮机和相关动力设备的首次大规模运行，最初有一些不可预见的问题需要解决。一旦这些问题得到解决，膜分离制氮机就会以所需的流速 24 小时连续运行，直到达到所需的氧气浓度。当顶层空间达到理想的氧气浓度后，关闭所有应用阀门，并对筒仓进行为期 11 天的监控。11 天后，实验终止，因为谷物需要装运，并在第 13 天装运完毕。

通常熏蒸后需通风散气，由于是用氮气进行处理，因此可以安全地打开粮仓和装运谷物，而不需要通风处理。

关键时间点和运行成本：

共用了约 116 h 的吹扫时间，以将顶空的氧气含量降至 1.6%；

仓内在 >97% 的氮气条件下可维持 270 h；

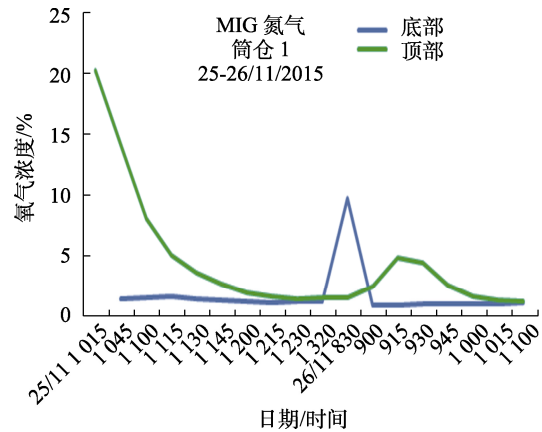
柴油发电机在 116 h 的吹扫过程中消耗了约 1 500 L 柴油（12.9 澳元/小时的柴油消耗成本）；

根据每升柴油 1.45 澳元的平均价格，处理的大致总运行成本为 2 175 澳元，即 99 美分/吨粮。

2.3 膜分离制氮技术实仓实验

从 1 号筒仓开始连续对筒仓进行吹扫。最初对 1 号筒仓的吹扫耗时 2.25 h，使氧气含量降至 1%。在此期间，氮气发生器的运行率为 98.8%。制氮机的出口管道在初始吹扫净化的后期发现了泄漏，这就是输出纯度低的原因。泄漏修复后，该装置在实验的剩余时间内输出的纯度为 99.3%。第二天，又对筒仓进行了 1.75 h 的吹扫。二次是对谷物中呼吸出来一晚上的氧气和二氧化碳进行吹扫。二次吹扫是氮气处理所需的最后一次“补充”。如果粮仓被确认为气密性良好，那么在暴露期内，氧气浓度可保持在 3% 以下（图 10 和 11）。

总施用时间为 4.5 h。为制氮系统提供动力的柴油发电机使用 11.7 L/h 柴油，根据 1.45 澳元/L 的柴油成本计算，筒仓 1 的处理总成本为 76 澳元。如果筒仓最大容量为 75 t 谷物，则处理成本为 1 澳元/t。

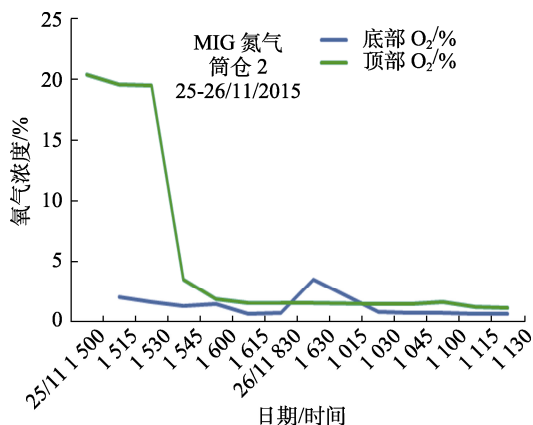


日期/时间	制氮机氮气 流速/(m ³ /hr)	椎体 O ₂ /%	顶部空间 O ₂ /%
11 月 25 日-10:15	30		20.2
10:45	33	1.2	14.1
11:00	33	1.3	7.9
11:15	33	1.4	4.9
11:30	33	1.2	3.4
11:45	30	1.1	2.5
12:00	30	1.0	1.7
12:15	30	0.9	1.4
12:30	30	1.0	1.2
13:20		1.0	1.3
11 月 26 日-08:30		9.6	1.3
09:00	27	0.7	2.2
09:15	30	0.7	4.7
09:30	30	0.8	4.2
09:45	33	0.8	2.4
10:00	33	0.8	1.4
10:15	33	0.8	1.1
11:00		0.9	1.0

图 10 Mingenew 实仓实验中 1 号筒仓应用氮气时
氧气浓度和氮气发生器流速数据

Fig.10 Oxygen concentration and N₂ generator flow rate data from N₂ application to Silo 1, Mingenew field evaluation site

2 号筒仓的初始吹扫净化时间为 1.5 h。由于制氮机的氮气流量增加，达到低氧水平的的时间缩短了。为实现快速吹扫，制氮机的流量增加到 60 m³/h 99% 的氮气。根据对 1 号筒仓的观察，可以知道流量的增加意味着泄压阀会迅速起泡。为了缓解这种情况，将泄压阀的盖子取下。这将减轻筒仓上的额外压力，并形成二级吹扫净化排气口。一旦观察到氧气衰减有明显的反应，就更换盖子，并关闭发生器，提供 30 m³/h 99.3% 的氮气（图 10 和 11）。第二天，对 2 号筒仓再进行 1 h 的吹扫，



日期/时间	制氮机氮气 流速/(m ³ /hr)	椎体 O ₂ /%	顶部空间 O ₂ /%
11月25日-15:00	35		20.4
15:15	60	2.0	19.6
15:30	60	1.6	19.5
15:45	53	1.3	3.5
16:00	53	1.4	1.8
16:15	30	0.6	1.5
16:30	30	0.7	1.5
11月26日-08:30		3.5	1.5
10:15			
10:30	33	0.8	1.4
10:45	33	0.7	1.4
11:00	33	0.7	1.6
11:15	29	0.6	1.2
11:30		0.6	1.1

图 11 Mingenev 实仓实验中 2 号筒仓应用氮气时氧气浓度和制氮机氮气流速数据

Fig.11 Oxygen concentration and N₂ generator flow rate data from N₂ application to Silo 2, Mingenev field evaluation site

以清除过夜呼吸的氧气和二氧化碳。这相当于 0.60 美元/t 的处理成本。由于各筒仓的装载量和粮龄存在明显差异，因此很难对充氮效率进行比较。1 号粮仓装的是上一季的谷物，与 2 号粮仓装的新季谷物相比，1 号粮仓吸附了更多的二氧化碳和氧气。这一点从隔夜的呼吸量可以明显看出，从而导致充氮时间延长。2 号粮仓装满了新季谷物，可以更可靠地估算运营成本。

两个粮仓都留出了一部分谷物样本，用于培养观察害虫自然感染的情况。第一周的初步计数表明，因筒仓 1 中谷物的年份和没有熏蒸历史，筒仓 1 中有 3 种昆虫，筒仓 2 中没有成虫(表 1)。检测到的害虫有谷蠹、锈赤扁谷盗和赤拟谷盗。

表 1 通过培养观察两个仓中害虫的情况，用于进一步评估操作效率

Table 1 Purging Observations and Potential to achieve further operation efficiency

第一周	虫种	1-1 仓	1-2 仓	2-1 仓	2-2 仓
	谷蠹	8	3	0	0
2015.3.12.	锈赤扁谷盗	1	1	0	0
	赤拟谷盗	4	0	0	0

用于本次实验的两个筒仓不相邻，因此放弃了将排出的富氮气吹扫到下一个筒仓的想法。虽然进行了短暂的实验，但由于距离过远产生了背压，增加的氮气流速对吹扫净化料仓造成了过大的压力，导致溢流阀剧烈起泡。事实证明，在关闭溢流阀盖对 2 号料仓进行吹扫时，提高氮气发生器的流速可大大缩短吹扫时间。这一观察结果表明，可以同时多个筒仓进行串联充气，但要注意控制对第一个料仓增加的压力。串联的筒仓应彼此相邻，以减少软管距离，筒仓之间应安装直径较大的排气软管，以释放减轻施加在第一个筒仓的压力。

3 讨论与结论

本次 PSA 制氮技术的实仓实验代表了一种“最坏情况”，即使用小产气量的制氮机来对容量较大的筒仓进行充气。例如，使用 30 m³/h PSA 发生器来处理几乎空仓的 500 t 筒仓，用 60 m³/h 膜分离制氮机来处理 2 300 t 的筒仓。事实证明，膜分离制氮机在处理实验所选筒仓这样的大型储存设施时非常高效。膜分离氮气发生器能够根据应用情况调节流速和纯度水平，大大缩短了吹扫净化筒仓大气所需的时间，从而降低了处理成本。与 PSA 氮气发生器相比，膜分离具有以下固有优势：

- 相同气体产量的系统占地面积更小；
 - 活动部件更少，意味着维修间隔更长；
 - 膜组件在正确的操作条件下使用寿命长达 20 年；
 - 噪音较小；
 - 压力容器无需持续认证。
- PSA 与膜分离在制氮和应用方面的比较：
膜分离制氮效率是 PSA 的两倍；
大产量膜分离制氮机的出现提高了与大容

量粮库相匹配的净化效率；

氮气气调技术可应用于 CBH 所有目前能够熏蒸的粮食仓库，控制昆虫和质量，价格比使用 VaperPh3os 磷化氢钢瓶气熏蒸便宜；

膜分离可以实现更高效的充氮和合适应用成本（表 2）。

表 2 制氮机产气量和筒仓容量与成本的关系
Table 2 The relationship between capacity of nitrogen generator and silo with cost

仓容(t)/产氮气粮(m ³ /hour)	成本(\$ per t)
2 300 t/60 m ³ /hour = 38 (膜分离制氮机)	\$0.99
2 300 t/120 m ³ /hour = 19 (膜分离制氮机)	\$0.84
500 t/30 m ³ /hour = 17 (PSA)	\$2.43
500 t/60 m ³ /hour = 9 (PSA)	\$2.15

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英文原文详见 P47-54。

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Delivery and Adoption of Nitrogen/Low Oxygen and Nitrogen + Phosphine Technology for the Management of Grain Storage Pests in Commercial Silos (英文原文)

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Abstract: Nitrogen technology has been developed as a complementary technology with fumigants, for control of key pests in Australian grain storages. However, market feedback from industry was its cost which remained as a barrier of uptake. Therefore, a commercial scale trial was set up using advanced membrane technology to assist industry overcome operational and the cost barriers for the uptake of the technology. A new generation membrane technology was sourced from Changshun Anda, a China-based company. The unit was deployed at the CBH grain port of Kwinana, Western Australia to compare performance against an older nitrogen generation technology (Pressure Swing Absorbance / PSA). To meet the strict Australian standards operating conditions the port the unit was upgraded by Changshun Anda specialist team. The target benchmark was set to \$0.50 per tonne of grain (the high-end cost of a phosphine fumigation). Research demonstrated an operational cost for the older PSA technology of \$2.43 per tonne of grain. The technology also displayed operational limitations in its ability to generate and maintain the required level of nitrogen purity necessary to provide insect control (99%). In comparison, the generation of nitrogen using membrane technology cost \$0.99 per tonne of grain. Other performance gains were generation of required nitrogen purity within 4.5 days of operation and ability to maintain purity for the required 14 days to provide full control of pests.

Key words: grain storage; stored grain insect management; nitrogen storage; Pressure Swing Adsorption (PSA) nitrogen generators; Membrane Separation (MS) nitrogen generators

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1 INTRODUCTION

Although phosphine is still the most commonly-used gas fumigant for controlling pests in stored grain, other options are required to be explored. One area of interest is the use of controlled atmospheres in which oxygen, carbon dioxide and nitrogen concentrations as well as temperature and humidity are regulated. In grains, legumes and oilseed the primary aim of the atmosphere is to control insect pests as most insects cannot exist indefinitely without oxygen. The technology has a long history in the industry^[1-3] and has known to be a fairly slow process, taking up to several weeks especially at lower temperatures (less than 15 °C).

In 2016, Australian grain industry established an investment project – Phosphine Alternatives to develop nitrogen technology as a complementary technology to the use of fumigants. The investment developed efficacy data and optimal nitrogen operating levels for control of key pests in Australian grain storages. The investment used pressure-swing absorption (PSA) technology to generate the required purity of nitrogen which is 99%. The technology is based on separation of some gas species from a mixture of gases using specific adsorptive materials (e.g., zeolites, activated carbon, and molecular sieves) as a trap, preferentially adsorbing the target gas species at high pressure. The process then swings to low pressure to desorb the adsorbed material. Industrial nitrogen generator units which employ the PSA technique produce high purity nitrogen gas (up to 99.9%) from a supply of compressed air. To resolve costs barriers and drive impact opportunities the investment from a delivery and impact perspective, the research included an on-farm trial (held in WA) which resulted in a positive financial result for the grower involved as he gained a market premium for the treated grain (based on an “organic” classification).

However, market feedback from industry was that cost remained a barrier to uptake; the need for gas-tight storages was another barrier although this is reducing as insect resistance to the fumigant

phosphine drives grain industry uptake of sealed storages. As a next step research has taken advantage of recent advances in the cost of nitrogen generation (membrane technology) by the mining industry to assist industry overcome operational barriers to uptake of the technology. Nitrogen membranes separate gases by the principle of selective permeation across the nitrogen membranes wall and have several operational advantages over PSA technology.

2 METHOD AND MATERIALS

2.1 Commercial trial of generation technology – PSA technology

Pressure Swing Absorbance technology was used on-site at several port terminals operated by CBH Australia. Trials were undertaken at the port of Kwinana where the mobile PSA generator that is on-site produces 99.5% pure nitrogen at 30 m³/hour. The silos selected at Kwinana terminal, Western Australia, were concrete vertical silos that have been sealed with a membrane to make them gas tight to achieve for general phosphine fumigation. The trial with PSA nitrogen generator was conducted in a 550 tonne ‘star’ cell approximately 20 tonne of barley. A ‘star’ cell is the void between four ‘main’ cylindrical cells created during construction. To boost storage capacity and provide additional grain segregation options at the terminal, these voids were sealed and used for the storage of grain.

The grain outlet chutes on the silos used for the trials were fitted with ball valves and 3” camlock fittings to facilitate nitrogen application. The silos are also fitted with internal piping to allow fumigant gas to be recirculated under normal fumigation events. The headspace exit pipe would be used as the purging location for the silos internal atmosphere. The headspace oxygen concentration would also be monitored at this location. The oxygen concentration values were recorded with a combination of Draeger XAM-7000’s and Toxipro personal protection monitors. The silo headspace and ambient temperature data was recorded via Onset Hobo temp/RH monitors.

2.2 Commercial trial of generation technology – membrane technology

The membrane separation nitrogen generator acquired by Murdoch University from Changshun Anda, China produces 60 m³ of 99.5% pure nitrogen. The system had undergone upgrades and certification to Australian electrical standards prior to the trials commencing. This has been done by contracted 3rd parties with the support and advice of specialists from Changshun Anda. The unit was also made fully portable / mobile to facilitate transfer of the unit between sites and within large commercial sites (such as the Kwinana port facility). Both the compressor and membrane generator were set on a truck. The truck then has an associated trailer which can transport the required power (a diesel generator) for when sites are unpowered or as a back up to existing power supply (Figure 1).



Fig.1 Mobile fumigation unit comprising compressor, membrane separation nitrogen generator and diesel generator outside grain storage at the Kwinana terminal.

The trial was conducted in a 2,300 tonne ‘main’ cylindrical cell, filled with approximately 2,200 tonne of barley. Larger capacity silo was used due to the unit having a higher nitrogen production capacity. The flow rates and nitrogen purity of the membrane units can be adjusted, giving either increased flow rates or increased nitrogen purity.

The system was capable of producing on average, 85m³ per hour of 98.5% pure nitrogen, after a brief warm up period. Initially increased flow rate at a lower purity was opted to enable a quick purge of the silo. Once a reasonable drop in oxygen concentration was observed, the flow rate was decreased to 40 m³ per hour to boost the nitrogen purity to 99.5%. The same setup and application procedure was undertaken for the membrane trial as the PSA trial. From the top of the silo internal atmosphere purging and monitoring was conducted. The pipe shown in the photo is connected to the headspace of the silo. This is the

location for the oxygen concentration monitoring. The ‘gate’ valve at concrete level was closed prior to the membrane generator being switched off to build slight positive pressure in the silo.

A Toxipro oxygen monitor was used as shown hanging at the top of the yellow safety cone. This would demonstrate that the exhausted depleted oxygen atmosphere posed no danger to staff walking past the fumigation area. In fact, CBH staff could only detect a drop from normal atmospheric oxygen content within 30 cm of the end of the pipe.

2.3 Field trial of generation technology – membrane technology

Field trials were conducted at Mingenew, WA grain zone in partnership with the Mingenew Irwin Group (MIG). The two silos chosen for the demonstration were modern 75 tonne elevated cone bottom silos fitted with factory installed ground level phosphine application, thermosiphon systems and pressure relief valves. The silos were first pressure tested to ensure suitability for a Nitrogen treatment. To conduct the pressure test, air pressure was applied to the silo with the aid of an air blower through the pressure relief valve. A digital handheld manometer was attached to observe a pressure increase of 250 Pa. With the air blower disconnected, the increase was timed until the pressure decayed to 125 Pa, giving us the silo’s ‘half-life’. Both silos returned a ‘half-life’ greater than 5 minutes, confirming their suitability.

One of the silos (Silo 1) contained 2016 Wheat and was 80% capacity. The other silo (Silo 2) contained new seasons Wheat and was 100% capacity. None of the grain had been treated previously with phosphine. A high level of Carbon Dioxide was observed in Silo 1, which could indicate the presence of grain insects. A grain sample was taken from both silos for its grain quality attributes and compared with an after-treatment grain sample at the completion of the trial. A portion of the grain samples were also placed in the controlled temperature culture facility at the Post Harvest Biosecurity laboratory, Murdoch University to assess further development of naturally infested grain insects, over a 5-week period.

Monitoring lines were installed to measure the silos Oxygen concentration at two locations, headspace and in the cone at the base of the silo. Temperature loggers were installed in the headspace of both silos and one was placed in the

environment to monitor ambient conditions. To facilitate the application of Nitrogen to the silo, a tap system was constructed to be installed into part of the silos thermosiphon circulation system. Gas tight UV stable ball valves were used to construct the application setup. Nitrogen was applied to the silo via a tap system installed in the pipe running down the cone at the base of the silo leading from the thermosiphon pipe to the phosphine fumigation chamber. The system and key operational activities are described in Figures 2~9.



Fig.2 Nitrogen application underway to 75 tonne silos. This Membrane Nitrogen generation unit (60 m³/hour, 99% pure nitrogen) is completely mobile being powered by a diesel generator.



Fig.3 Nitrogen is produced via 60 m³/hr, 99% purity Membrane separation Nitrogen Generator. Hose of diameter 3/4" connected to outlet.



Fig.4 Tap system installed in existing thermosiphon pipework. Thermosiphon pipework consists of 90 mm stormwater PVC pipe leading from headspace of silo to ground level phosphine application chamber. Phosphine chamber has entry duct into base of silo, to complete the recirculation loop.



Fig.5 At the join of the cone and sidewall of the silo, the thermosiphon pipe reduces in diameter to 40 mm Drain and Waste (DNW) pipe. This reduced diameter pipe connects with the ground level phosphine application chamber. It is this pipe where the Nitrogen application tap system was installed into.



Fig.6 Nitrogen is applied to the silo via tap system incorporating UV stable ball valves and camlock fittings for ease of hose connection.



Fig.7 A length (550 mm) of the 40 mm pipe was cut and removed to facilitate installation of the tap system. A brace was installed to support the pipe given the additional weight of the tap system. Two 40 mm rubber sleeves were used to connect onto the 40 mm pipe. Four hose clamps were installed and fastened to complete the gas tight installation.

Nitrogen is applied to the silo with the inloading lid locked in the closed position. The silos internal atmosphere is displaced and purged out of the silo via the thermosiphon pipe. It is exhausted at ground level via the ball valve closest to the wall of the silo.

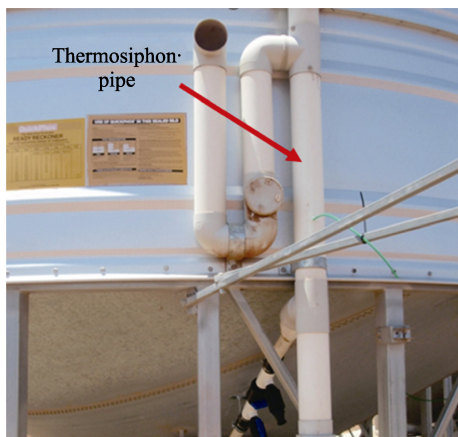


Fig.8 Once the Nitrogen application system had been installed, a pressure test was conducted to ensure the silo was gas tight enough to maintain adequate concentrations of N_2 . Silos sold as gas tight and fitted with thermosiphon systems require pressure relief valves for compensation of temperature fluctuations to prevent silo damage. Pressure tests are conducted by applying a volume of air (250 Pa) to the silo and with a handheld manometer and stopwatch, timing the decay of that applied pressure to half (125 Pa) giving its 'half loss time'. A value in minutes. The silos for these trials returned a 'half loss time' of 5 minutes indicating suitability for the application of Nitrogen.



Fig.9 Once the Oxygen concentration in the Headspace and cone of the silo return values of 1% or less, indicating the presence of 99% Nitrogen, the Nitrogen production can be reduced and the exhaust ball valve closed. Briefly continuing a low flow (around $10 \text{ m}^3/\text{hour}$) of Nitrogen will build a slight positive pressure in the silo, which will aid in the silos ability to maintain high concentrations of Nitrogen. The N_2 application ball valve is then closed and hose withdrawn. The hose can be unfastened whilst still flowing Nitrogen as the flow is minimal. The inline ball valve is then opened allowing Nitrogen to circulate around the silo via the thermosiphon system, as dictated by the daily temperature fluctuations.

This system of application enables the Nitrogen to be purged into the base of the silo. This is achieved by closing the in-line tap and directing the generated Nitrogen into the base of the silo, via the fumigation chamber. The fumigation chamber has an outlet connected to the base of the silo completing the loop from the thermosiphon system.

Nitrogen then percolates through the grain bulk displacing the internal atmosphere. Nitrogen is applied with the silo lid locked down, this directs the purged atmosphere out of the top of the silo via the thermosiphon pipe. The atmosphere is then exhausted at ground level via another ball valve tap installed adjacent to the application port.

The Membrane separation generator was set to deliver 33 m^3 per hour of 99.4% pure Nitrogen. A higher flow rate was initially set, as this particular unit can deliver up to 60 m^3 of 99% Nitrogen, however in this case the higher flow caused the pressure relief valves to bubble quite rapidly. At 33 m^3 per hour the relief valve bubbled gently. Nitrogen was delivered to the silo via a 20 mm diameter braided hose, fitted with $\frac{3}{4}$ " camlock fittings. The silos headspace and cone were monitored throughout the Nitrogen application period to determine when the application could cease. Application continued until the Headspace and cone returned oxygen values of 1% indicating 99% Nitrogen.

At the completion of the application period, the atmosphere exhaust ball valve was closed first, building a slight positive pressure in the silo. The application port was then closed and the hose withdrawn. To enable the thermosiphon system to function correctly again, the central in-line tap was then opened allowing Nitrogen to be circulated around the silo dictated by the daily temperature fluctuations.

3 RESULTS

3.1 Commercial trial of generation technology – PSA technology

Key findings are:

The cell took approximately 84 hours for the oxygen concentration to drop below 5%;

It took a further 84 hours to reduce to 3% oxygen;

The cell was monitored for a further 3 days, but the Oxygen concentration had stabilized at 3.3%. No further reduction had been observed;

Total application time was of approximately 168 hours.

The PSA nitrogen generator is mains powered unit, which makes it quite difficult to obtain energy consumption figures for calculation of a treatment cost. However, based on Lake Grace grain grower PSA nitrogen purging trials use of same model and

capacity PSA nitrogen generator, diesel generator consumed approximately 5 litres of diesel per hour purging and an average price of \$1.45 per litre of diesel, approximate total running cost of the treatment was \$1,218 or \$2.43 per tonne.

3.2 Commercial trial of generation technology – membrane technology

As this was the first major run for the membrane generator and associated power equipment, there were some initial unforeseen issues that required solving. Once these issues were rectified, the membrane generator was running at the desired flow rate continuously, 24 hours a day, until a satisfactory level of oxygen was reached. Once the desired level of oxygen had been reached in the headspace, all application valves were closed and the silo monitored for a further 11 days. The trial was terminated after 11 days as the grain was requested for shipping and out loaded on the 13th day.

Given that the treatment was conducted with nitrogen, the silo could be opened and the grain out loaded safely and without the withholding periods normally associated with venting fumigants.

Key timing and price observations are:

Total application time of approximately 116 hours of purging to reduce the oxygen content to 1.6% in the headspace;

Cell maintained for a further 270 hours at > 97% nitrogen;

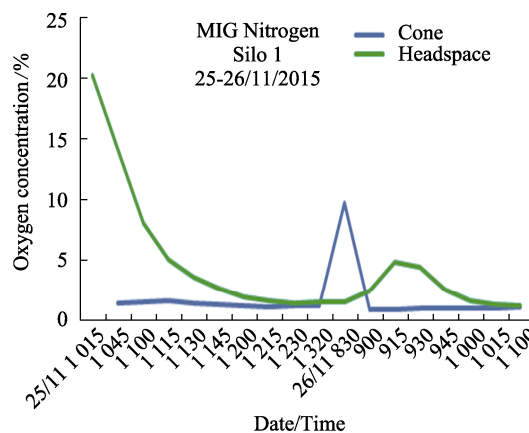
Diesel generator consumed approximately 1,500 litres of Diesel over the 116 hours of purging (\$12.9/hour diesel consumption cost);

Approximate total running cost of the treatment was \$2,175 or 99 cents per tonne, based on an average price of \$1.45 per litre of diesel.

3.3 Field trial of generation technology – membrane technology

The silos were purged consecutively beginning with Silo 1. The initial purge of Silo 1 took 2.25 hours to bring the Oxygen level down to 1%. The N₂ generator was running at 98.8% over this period. A leak in the outlet pipe of the Nitrogen generator was detected late into this initial purge which would account for the low output purity. After the leak was fixed the unit would deliver 99.3% for the rest of the trial. The following day, the silo was purged for another 1¼ hrs. The secondary application purges all the additional oxygen and carbon dioxide respired from the grain overnight. This secondary purge is the final ‘top-up’ required for a Nitrogen treatment.

If the silo is confirmed as gas tight, the oxygen concentration can be maintained below 3% for the duration of the exposure period (Figure 10 and 11).

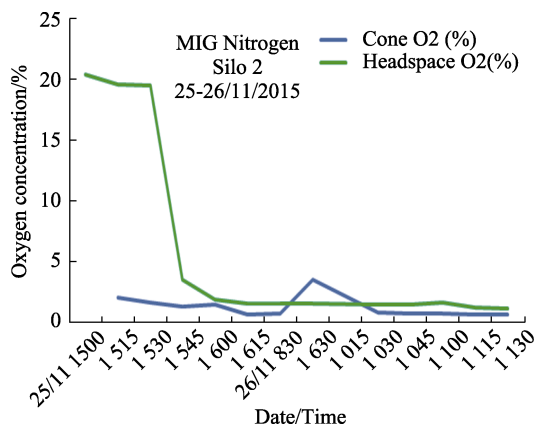


Date/Time	N ₂ Generator Flow Rate/(m ³ /hr)	Cone O ₂ /%	Headspace O ₂ /%
25/11-10:15	30		20.2
10:45	33	1.2	14.1
11:00	33	1.3	7.9
11:15	33	1.4	4.9
11:30	33	1.2	3.4
11:45	30	1.1	2.5
12:00	30	1.0	1.7
12:15	30	0.9	1.4
12:30	30	1.0	1.2
13:20		1.0	1.3
26/11-08:30		9.6	1.3
09:00	27	0.7	2.2
09:15	30	0.7	4.7
09:30	30	0.8	4.2
09:45	33	0.8	2.4
10:00	33	0.8	1.4
10:15	33	0.8	1.1
11:00		0.9	1.0

Fig.10 Oxygen concentration and N₂ generator flow rate data from N₂ application to Silo 1, Mingenew field evaluation site

Total application was 4.5 hours. The diesel generator powering the Nitrogen generation system used 11.7 L per hour, indicating a total cost for the treatment of Silo 1 at \$76, based on a diesel cost of \$1.45 per litre. If the silo held the maximum capacity of 75 tonnes of grain, this equates to a treatment cost of \$1 per tonne.

The initial purge of Silo 2 took 1.5 hours. The reduced time to low oxygen levels was due to an increased flow rate of Nitrogen from the generator. The generator was wound up to deliver 60m³ of



Date/Time	N ₂ Generator Flow Rate/(m ³ /hr)	Cone O ₂ /%	Headspace O ₂ /%
25/11-15:00	35		20.4
15:15	60	2.0	19.6
15:30	60	1.6	19.5
15:45	53	1.3	3.5
16:00	53	1.4	1.8
16:15	30	0.6	1.5
16:30	30	0.7	1.5
26/11-08:30		3.5	1.5
10:15			
10:30	33	0.8	1.4
10:45	33	0.7	1.4
11:00	33	0.7	1.6
11:15	29	0.6	1.2
11:30		0.6	1.1

Fig.11 Oxygen concentration and N₂ generator flow rate data from N₂ application to Silo 2, Mingenev field evaluation site

99% Nitrogen to achieve a quick purge. Based on observations from Silo 1, it was known that an increase in flow rate would mean a rapid bubbling of the pressure relief valve. To alleviate this, the cap was removed from the pressure relief valve. This would relieve the additional pressure on the silo and create a secondary purging exhaust port. Once a significant response was observed in the oxygen decay, the cap was replaced, and the generator wound down to deliver 30 m³ per hour of 99.3% Nitrogen (Figure 10 and 11). The following day Silo 2 was purged for an additional 1 hour to purge the respired oxygen and carbon dioxide overnight. Total application was 2.5 hours, indicating an operation cost of \$43. This equates to a treatment cost of \$0.60 per tonne. Given the clear differences in fill capacity and grain age between the silos, it is difficult to compare the application efficiencies. Silo 1 contained last season's grain which retained more carbon dioxide and oxygen as new season's grain in Silo 2. This is evident by the overnight respired levels, leading to an extended

application period. Silo 2, having a full silo of new season's grain would provide a more reliable estimation of operation costs.

A portion of the grain samples from both silos have been set aside for the culture of naturally infested insects. An initial Week 1 count indicated 3 species of insects in Silo 1 and nil adults present in Silo 2. This is to be expected given the age of grain in Silo 1 and no fumigation history (Table 1). Insects detected were *Rhyzopertha dominica* (Lesser Grain Borer), *Cryptolestes ferrugineus* (Rusty Grain Beetle) and *Tribolium castaneum* (Red Flour Beetle).

Table 1 Purging Observations and Potential to achieve further operation efficiency

Week 1	species	Silo 1-1	Silo 1-2	Silo 2-1	Silo 2-2
3.12.15	<i>Rhyzopertha dominica</i>	8	3	0	0
	<i>Cryptolestes ferrugineus</i>	1	1	0	0
	<i>Tribolium castaneum</i>	4	0	0	0

The Silos made available for the trial were not adjacent leading to the idea of purging exhausted Nitrogen rich atmosphere into the next silo being abandoned. It was trialled briefly but due to the back pressure created over distance, the increased N₂ flow rate put too much pressure on the purge silo, causing the relief valve to bubble violently. Increasing the flow rate of the Nitrogen generator whilst purging Silo 2 with the relief valve cap off proved to reduce the purging time significantly. This observation indicates that it is possible to purge multiple silos simultaneously, with attention given to managing the increased pressure placed on the first 'charge' silo. Silos to be linked should be adjacent to one another to reduce the hose distance, and larger diameter exhaust discharge hoses between the silos should be installed to relieve the pressure applied to the first 'charge' silo.

4 DISCUSSION AND CONCLUSION

The field trial of generation technology – PSA technology represented a “worst case scenario” with small capacity nitrogen generators for larger silos eg, PSA generator (30 m³/hour) for almost empty silo with 500 tonne capacity and 60 m³/hour membrane separation nitrogen generator for 2,300 tonne silo, which significantly result to take longer purging period to overcome gas mixing. The

membrane separation nitrogen generator has proven to be very efficient in treating such a large storage facility as the silo chosen for the trials. The ability to adjust the flow rates and purity levels to suit the application has significantly reduced the time taken to purge the silo's atmosphere, thereby reducing the cost of the treatment. When compared with PSA nitrogen generators, the membrane generators have the following inherent advantages:

Larger capacity system on a smaller footprint;

Less moving parts, means longer service intervals;

Membrane modules have a service life of 20 years under the correct operating conditions;

Membrane nitrogen generator being less noisy;

No pressure vessels which require ongoing certification.

Comparison between PSA generator and membrane nitrogen generator for generation and application of nitrogen:

Membrane nitrogen generator got double the efficiency over PSA;

The availability of larger capacity membrane nitrogen generators improved the purging efficiency when matched to large capacity grain storages;

Nitrogen technology can be applied to all current CBH grain storages that are capable of fumigation to achieve insects and quality control with price cheaper than fumigation with VaperPh3os;

To achieve higher efficient nitrogen purging and cost (Table 2).

Table 2 The relationship between capacity of nitrogen generator and silo with cost

Silo capacity (tonne) / N ₂ generator capacity (m ³ /hour)	Cost (\$ per tonne)
2 300 tonne / 60 m ³ /hour = 38 (Membrane nitrogen generator)	\$0.99
2 300 tonne / 120 m ³ /hour = 19 (Membrane nitrogen generator)	\$0.84
500 tonne / 30 m ³ /hour = 17 (PSA)	\$2.43
500 tonne / 60 m ³ /hour = 9 (PSA)	\$2.15

REFERENCES

See in its Chinese version P46. 