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Biofiltration of *n*-butyric acid for the control of odour

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Abstract

Odour control from pig production facilities is a significant concern due to increased public awareness and the development of more stringent legislation to control production. Although many technologies exist, biofiltration is still the most attractive due to its low maintenance and operating costs. One of the key odour components *n*-butyric acids was selected for a laboratory scale biofilter study. It was examined as a sole carbon substrate in order to investigate the effectiveness of biofiltration in reducing *n*-butyric acid concentration under different operating conditions using a moist enriched woodchip medium. Three superficial gas velocities; 38.2 m h⁻¹, 76.4 m h⁻¹, and 114.6 m h⁻¹ were tested for *n*-butyric acid concentrations ranging from 0.13 to 3.1 g [*n*-butyric acid] m⁻³ [air]. For superficial gas velocities 38.2 m h⁻¹, 76.4 m h⁻¹, and 114.6 m h⁻¹, maximum elimination capacities (100% removal) of 148, 113 and 34.4 g m³ h⁻¹, respectively, were achieved. Upon investigation of effective bed height, true elimination capacities (100% removal) of 230, 233 and 103 g m⁻³ h⁻¹, respectively, were achieved at these superficial gas velocities. Averaged pressure drops for superficial gas velocities 38.2 m h⁻¹, 76.4 m h⁻¹, and 114.6 m h⁻¹ were 30, 78 and 120 Pa, respectively. It was concluded that biofiltration is a viable technology for the removal of *n*-butyric acid from waste exhaust air, but near 100% removal efficiency is required due to the low odour detection threshold for this gaseous compound.

Keywords: volatile fatty acid; butyric acid; odour; biofilter; olfactometry; pigs

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1. Introduction

Intensive pig production can cause odorous gases to be formed and their release can cause nuisance in the vicinity of such facilities. An increase in public awareness has led to the stimulation of development of better legislation to control their release. The main sources of these odorous gases include building ventilation air, manure storage facilities and the spreading of manure. Often these odorous mixtures are a result of animal manure decomposing anaerobically to form unstable intermediate by-products resulting in a complex mixture of over 168 volatile compounds of which 30 are odorous (O'Neill and Phillips, 1992). These compounds resulting from natural biological reactions include aldehydes, alcohols, fixed gases (methane, carbon dioxide, etc.), carbonyls, esters, amines, sulphides, mercaptans, aromatics, nitrogen heterocycles and volatile organic acids at concentrations ranging from near 0 to 10 mg [Volatile Fatty Acids] m⁻³ [air] (Le Cloirec et al., 1988; Tanaka et al., 1991).

Butyrate (butyric acid ion in solution) is one of the major intermediates in the anaerobic degradation of organic compounds (Fang et al., 1995). This mechanism is known as acidogenic fermentation (Mainville et al., 1996). Studies performed by Williams (1994), Zhu et al. (1998) and Baumgartner Environics Inc. (2000) indicate that volatile fatty acids are major indicators of pig odour offensiveness.

Hartung (1987) suggested that up to 617 µg [*n*-butyric acid] m⁻³ [air] could be emitted by way of the exhaust ventilation air and that *n*-butyric acid is the major volatile organic acid (Anon, 2000). Martensson et al. (1997) reported an average concentration of 760 µg [*n*-butyric acid] m⁻³ [air] in the ventilation air of an intensive, partially slatted, pig building. Smith et al. (2000) documented average concentrations of 4990 µg [butanoic acid] m⁻³ [air] and 377 µg [butanoic acid] m⁻³ [air] present in the headspace above pig faecal and urine samples, respectively.

Even at these low concentrations, volatile fatty acids (VFA) have high odour nuisance values and can be detected at several kilometres from the facilities (Ali et al., 2000). *n*-Butyric acid has a distinctive odour (i.e. sweet rancid) with a low odour threshold value of 0.4 to 3.6 μg [*n*-butyric acid] m^{-3} [air] (O'Neill & Phillips, 1992; Tamminga, 1992), and hence contributes significantly to the odour threshold concentration and intensity of pig odour. In accordance with Japanese offensive odour control laws, *n*-butyric acid is 30 and 35 times more offensive than hydrogen sulphide and propionic acid, respectively, at an odour intensity of 3 (Tanaka, 2000).

Although many technologies exist for odour control, biofiltration is still one of the most attractive due to its low maintenance and operating costs. It is a proven technology as a method of reducing emissions of odour and volatile organic compounds from industrial and commercial sources (Pearson et al., 1992; Goldstein, 1996; Vaith et al., 1996; Nicolai and Janni, 2000), but only limited information is available on the biofiltration of volatile fatty acids such as *n*-butyric acid (Mainville, 1996; Devanny et al., 1999).

The objective of this study was to investigate the effectiveness of a lab-scale biofilter in reducing *n*-butyric acid under different operating conditions using a moist enriched woodchip medium.

2. Methods

2.1. Biofilter apparatus

The laboratory scale biofilter used is shown in Fig. 1. The biofilter was comprised of a 1.2 m long, 100 mm internal diameter, glass cylinder, sealed at each end with stainless steel plates and Teflon seals. The effective packing height used was 800 mm, therefore allowing entry and exit spaces of 200 mm each in height. Gas sample ports were located at 0 mm (inlet), 125 mm, 250 mm, 375 mm, 500 mm, 625 mm, 750 mm and 800 mm (outlet) of packing height. These ports allowed for samples to be taken along the packing height of 800 mm to determine the penetration

profile of *n*-butyric acid through the medium. The filter was operated in down-flow mode to allow easy addition of water to the medium bed because air with a relative humidity of less than 100% can result in rapid loss of biodegradation activity, as the incoming gas steadily removes water from the bed by convection and dries out the biofilm first (Auria et al., 1998; Bohn and Bohn, 1999; Krailis et al., 2000).

The packing was supported on a perforated stainless steel plate. In addition to the gas inlet, the top plate was fitted with a port for liquid addition while the bottom plate was fitted with a gas outlet and a liquid drain. The temperature of the column was maintained between 23 and 27°C using a thermocouple method and heating tape (Tawil and Hamer, 2000). The gas flow rate through the biofilter was measured and controlled using a rotameter. The pressure drop in the filter bed was measured in-line using a differential pressure gauge (Dwyer, Manotherm, Dublin 12).

The main air supply to the biofilter was humidified with water vapour by sparging it through a temperature-controlled 25 l bottle containing deionised water. One minor air stream was sparged into a 1 l temperature-controlled bottle containing neat *n*-butyric acid and subsequently mixed with the major air stream. This mixed air stream was passed through a mixing chamber. The desired experimental concentrations of *n*-butyric acid were obtained by mixing various volumes of vapour-laden air with the humidified air prior to entry into the biofilter column.

2.2. Nutrient mineral salts medium

To develop an enrichment culture, the following sterile buffered mineral salts medium was made up: (g l⁻¹ deionised water); 1.4 g Na₂HPO₄·2H₂O, 0.7 g KH₂PO₄, 2.0 g (NH₄)₂SO₄, 0.6 g MgSO₄·7H₂O, 0.1 g CaCl₂·2H₂O, 0.1 g NaCl, 0.1 g EDTA and 1 ml l⁻¹ trace element solution as developed according to Pfenning et al. (1981). The pH of the medium was 6.8 (Tawil and Hamer, 2000).

2.3. *Inoculum*

The source of the microbial consortium used for the degradation of *n*-butyric acid was liquid from under a diesel storage tank area. Aerobic *n*-butyric acid degrading enrichment cultures were established in agitated shake flasks at 28°C with regular transfers every 7 days over an 8-week period. The growth medium used was a defined buffered mineral salts medium of pH 6.8, with *n*-butyric acid as the sole carbon energy substrate. The shake flasks contained 200 ml liquid volume and were closed with foam bungs to maintain oxygen transfer in the headspace of the conical flask. Enrichment cultures that developed significant visual turbidity were serially transferred to fresh sterile mineral salts medium containing 0.5 g l⁻¹ *n*-butyric acid until a mixed consortium exhibiting stable growth characteristics resulted.

When a stable mixed microbial consortium was established, 200 ml of the shake flask culture was added to 2800 ml of fresh sterile mineral salts medium in a bioreactor. Agitation was provided by means of a gas inducing impeller, which allowed recirculation of headspace gas. The bioreactor was equipped with controls for impeller speed, temperature and pH. It was operated at an impeller speed of 750 rpm, a temperature of 28°C and a pH of 6.8. This stable mixed microbial consortium was used to inoculate the moist enriched wood chip medium.

2.4. *Enriched packing medium*

The packing utilised was soft wood chip with an effective size of 5.6 mm to 10 mm. The dry packing density was 180 kg m⁻³. The voidage of the packing material was 62% and was calculated using the relationship proposed by Hodge and Devanny (1995). The average specific surface area of the packing material was approximately 292 m² m⁻³ (Bibeau et al., 2000). Prior to use, the packing material was mixed with 10 % w/w calcium carbonate for pH control and

saturated to 70% (w.w.b.) with sterile mineral salts growth medium (*Section 2.2*). This enriched wood chip medium was then mixed with 3 l of concentrated inoculum (*Section 2.3*), before packing uniformly into the glass column (Tawil and Hamer, 2000).

The moisture content (w.w.b.) of the enriched wood chip medium was intermittently checked by taking solid samples from the top, middle and bottom of the column; the samples were weighed before and after drying in an oven at 105°C for 48 hours. Water was added to the top of the biofilter as required to maintain a moisture content of 70% (w.w.b.). Samples of biofilter leachate (when available) and supply water were taken for pH measurement every day. Three pH buffer solutions; four, seven and nine were used to calibrate a Russell combination pH electrode which was linked to an Orion 520A pH/mV meter. An electrode slope was produced and the pH of each of the samples was determined.

2.5. Operating conditions

The total bed volume of the biofilter was 0.00628 m³. Downward gas flow rates ranging from 0.3 m³ h⁻¹ to 0.9 m³ h⁻¹ were used over the experimental period of 120 days. The volumetric loading rates on the filter were 47 to 143 m³ [gas] m⁻³ [medium] h⁻¹ and the empty bed residence times ranged from 46 to 15 seconds. The inlet gas to the filter had a relative humidity greater than 98%, which was checked regularly using a Testo 400 handheld temperature/relative humidity probe (Testo, UK). An IR CO₂ probe was used to confirm the production of carbon dioxide (Testo, UK). The initial inlet *n*-butyric acid concentrations ranged from 0.13 to 3.10 g [*n*-butyric acid] m⁻³ [air] in order to determine the degradation profile at different residence times and specific bed heights. The inlet concentration was changed every 4 days, after which time no change could be observed in the degradation profile of the biofilter bed.

2.6. Gas Chromatography

Gas phase concentrations of the inlet, outlet and intermediate ports of the biofilter were measured on-line using a gas chromatograph (GC) (Varian 3600, JVA, Dublin). The inlet, outlet and intermediate ports were sampled in triplicate using a sampling/injection apparatus consisting of a 2 ml sampling loop, 6 port switching valve (Valco Instruments Co. Inc., JVA, Dublin) and a vacuum pump. Both the valve and sample loop were heated to prevent condensation of saturated gas streams in the sampling line. The GC was equipped with a 2 m Haysep Q SS 80/100 packed column and was operated isothermally at a column temperature of 180⁰C with an injector temperature of 210⁰C and a detector temperature of 250⁰C. Peak area integration was performed using the software package Star chromatography Ver. 5 (JVA, Dublin).

2.7. Olfactometry

In order to collect air samples for olfactometry analysis, a static sampling method was used where air samples were collected in 8.5 litre Nalophan bags using a vacuum sampling device that operates on the 'lung principle', whereby the air is removed from a rigid container around the bag by a battery powered vacuum pump. This was performed according to Sheridan et al. (2002a).

An ECOMA TO7 dynamic yes/no olfactometer was used throughout the experimental period to determine the odour threshold concentration of the influent and outlet air of the laboratory scale biofilter. The olfactometry was performed according to Sheridan et al. (2002a).

2.8. Microbial analysis

In order to identify the bacterial genera responsible for the biodegradation of *n*-butyric acid, loopfuls of shake flask culture were streaked onto plate count agar and incubated at both room temperature and at 30⁰C. Five distinct colonies were noted and cultured in pure form. Each pure

culture was tested as follows: gram reaction, spore stain, catalase activity, oxidase activity and API test (Collins and Lyne, 1985).

3. Results and discussion

3.1. Physical sorption of n-butyric acid on to moist wood chip medium

A physical sorption experiment was undertaken to determine the sorption pattern that occurred when the vapour-laden gas was in contact with a moist sterilised wood chip medium. Pre-humidified air at a flow rate of $0.3 \text{ m}^3 \text{ h}^{-1}$ and an inlet concentration of $0.35 \text{ g [n-butyric acid] m}^{-3}$ [air] was contacted with the sterilized un-inoculated medium.

The outlet gaseous concentration of *n*-butyric acid asymptotically approached the inlet *n*-butyric acid concentration after approximately four hours in operation. The equilibrium mass of sorbed *n*-butyric acid was $1.26 \text{ g [n-butyric acid]}$, which corresponded to an interstitial water concentration of $1.59 \text{ g [n-butyric acid] l}^{-1}$ [water]. This is high when compared to water miscible solvents such as *n*-proponal (i.e. $0.12 \text{ g [n-proponal] l}^{-1}$ [water] communicated by Tawil (2001)). This may be explained by the high Henrys constant (i.e. 5.8×10^{-2}) of *n*-butyric acid and hence high affinity for water, assuming no sorption or interaction effects with the wood chip medium. Therefore, the moist sterilised wood chip medium could only remove $1.26 \text{ g [n-butyric acid]}$ by way of sorption, so any additional *n*-butyric acid loading was removed by means of microbial activity when using the inoculated medium.

3.2. Removal of n-butyric acid during steady state operation

The biofiltration of *n*-butyric acid emissions was carried out over 120 days at gas flow rates of 0.3, 0.6, and $0.9 \text{ m}^3 \text{ h}^{-1}$. This corresponded to empty bed residence times of 46, 23 and 15

seconds, respectively. The inlet *n*-butyric acid concentrations also varied from 0.13 to 3.1 g [*n*-butyric acid] m⁻³ [air]. The pH of the biofilter leachate ranged from 5.3 to 6.8.

During the 120-day experiment, outlet *n*-butyric acid concentration was recorded. Outlet concentration remained non-detectable until day 59, when the outlet concentration increased to 0.06 g [*n*-butyric acid] m⁻³ [air] and again to a maximum of 0.1 g [*n*-butyric acid] m⁻³ [air] on day 63. 200 ml of twice strength of nutrient mineral salts medium (*Section 2.2*) was added to the packing medium on day 59 and again on day 65. On day 69, the outlet *n*-butyric acid concentration returned to being non-detectable. Mainville (1996) reported that nitrate and other bioavailable forms of nitrogen levels decreased in a biofilter treating *n*-butyric acid. Highest consumption was encountered in the top section where highest inlet loading occurred. This suggested that immobilisation of inorganic nitrogen seemed to provide nutrients for the microorganisms responsible for *n*-butyric acid degradation. Gribbins and Loehr (1998) reported that soluble nitrogen concentration in the medium can limit biofilter performance after long periods of operation and even at low inlet volatile organic compound loading rates, the biofilter requires a threshold amount of soluble nitrogen to maintain pseudo steady-state operation. The addition of nitrogen in an available form allowed the biofilter to return to maximum elimination capacity. On day 106, *n*-butyric acid concentration was detected in the outlet stream of the biofilter. The addition of 200 ml of twice strength of nutrient mineral salts medium had no effect on the outlet concentration. This indicated that the biofilter had obtained maximum elimination capacity at this volumetric loading rate, assuming no other phenomenon was involved.

The maximum elimination capacities (100% removal efficiency) of *n*-butyric acid for air flow rates 0.3, 0.6 and 0.9 m³ [air] h⁻¹ were 148, 113.7 and 34 g [*n*-butyric acid] m⁻³ [air] h⁻¹, respectively. Complete removal was obtained at air flow rates of 0.3 and 0.6 m³ h⁻¹, but removal efficiency was reduced by 14% at an air flow rate of 0.9 m³ [air] h⁻¹ at a maximum inlet concentration of 0.54 g [*n*-butyric acid] m⁻³ [air]. Mainville (1996) obtained a maximum elimination capacity (97.5 % removal efficiency) of 29.14 g [*n*-butyric acid] m⁻³ [air] h⁻¹ using a

compost medium with average moisture content of approximately 50%. Eitner (1989) achieved a maximum elimination capacity (removal efficiency not specified) of $30 \text{ g } [n\text{-butyric acid}] \text{ m}^{-3} [\text{air}] \text{ h}^{-1}$ using a compost medium (Devinny et al., 1999). It is important to note that when *n*-butyric acid breakthrough occurred, removal efficiency decreased rapidly. Mainville (1996) suggested that inlet concentration not contact time was the principle factor in determining volumetric mass removal rate per volume of media. The confirmation of the production of CO_2 using an IR probe (i.e. 2.4 g m^{-3} of CO_2 corresponding to an inlet concentration of 3.1 g m^{-3} of *n*-butyric acid) in the outlet air stream from the biofilter demonstrated that microbial activity was the main mechanism for the removal of *n*-butyric acid.

3.3. Influence of airflow rate on the biodegradation along the bed height profile

One of the aims of this experiment was to determine the local *n*-butyric acid concentration along the packing bed height. The biofilter was operated in down flow mode, so the highest local concentration of *n*-butyric acid was at the top of the bed and the lowest local concentration of *n*-butyric acid at the bottom of the bed. Three volumetric loading rates of 47, 95.5 and $143 \text{ m}^3 [\text{air}] \text{ m}^{-3} [\text{medium}] \text{ h}^{-1}$ were examined, corresponding to empty bed residence times of 46, 23 and 15 seconds, respectively. It is important to note that the localised concentration profile provided valuable information on the degradation profile of the medium bed.

For a volumetric loading rate of $47 \text{ m}^3 [\text{air}] \text{ m}^{-3} [\text{medium}] \text{ h}^{-1}$, and an inlet *n*-butyric acid concentration of $3.1 \text{ g } [n\text{-butyric acid}] \text{ m}^{-3} [\text{air}]$, a bed height of 0.515 m was required for complete bio-oxidation of *n*-butyric acid, which equated to a true elimination capacity of $230 \text{ g } [n\text{-butyric acid}] \text{ m}^{-3} [\text{air}] \text{ h}^{-1}$.

For a volumetric loading rate of $95.5 \text{ m}^3 [\text{air}] \text{ m}^{-3} [\text{medium}] \text{ h}^{-1}$, the removal efficiency was decreasing with each stepped increase in inlet *n*-butyric acid concentration. It was thought that this might be due to nutrient deficiency, so 200 ml of twice strength nutrient mineral salts

medium was added and the removal efficiency recovered. An inlet *n*-butyric acid concentration of 1.19 g [*n*-butyric acid] m⁻³ [air] required a bed height of 0.39 m for complete bio-oxidation of *n*-butyric acid, which implied that the true elimination capacity was 233 g [*n*-butyric acid] m⁻³ [air] h⁻¹.

For a volumetric loading rate of 143 m³ [air] m⁻³ [medium] h⁻¹, the addition of nutrients to the medium bed did not affect the degradation profile of the medium. Complete bio-oxidation of inlet *n*-butyric acid concentrations of 0.13 and 0.24 g [*n*-butyric acid] m⁻³ [air] required a bed height of 0.265 m, which equated to a true elimination capacity of 103 g [*n*-butyric acid] m⁻³ [air] h⁻¹.

3.4. Microbial identification

n-Butyric acid is a relatively easy substrate to bio-oxidise (Devinny et al., 1999), so isolating a mixed microbial consortium was not difficult. Five different bacterial genera were isolated in accordance with the testing methodology in *Section 2.8*. They were all gram negative, catalase positive, non-spore forming rods/cocci. Two were identified as members of the genus *Enterobacter*; two as members of the genus *Moraxella*; and one was identified as a member of the genus *Pseudomonas*. Two fungal genera were isolated also but further identification was not performed (Quilty, 2001).

An important point to note is that the isolated microbial genera were only gamma-Proteobacteria. These are fast growing prokaryotes, which can overgrow the important microorganisms of the biofilter. The bacterial genera *Moraxella* and *Enterobacter* have not been isolated from a biofilter previously. Strains of the genus *Pseudomonas* are frequently isolated from biofilters, and observing the details from where the bacteria were isolated (i.e. under a diesel storage tank), it is not surprising this genus was present (Lipski, 2002). The presence of fungi within the biofilter bed could be explained by the acidic nature of the compound being bio-oxidised as fungi

can generally function in more harsh conditions (low pH and/or low water activity) than bacteria (Woertz and Kinney, 2000).

3.5. Implications for large scale biofilters

In accordance with the results obtained in this study, it would require empty bed true retention times of 30, 11 and 5 seconds in order to completely degrade inlet *n*-butyric acid concentrations of 3.1, 1.19 and 0.24 g [*n*-butyric acid] m⁻³ [air], respectively. The total combined maximum concentration of volatile fatty acids in the exhaust ventilation air of pig production buildings is approximately 0.08 g [VFA] m⁻³ [air] (Hartung, 1987), therefore a wood chip biofilter should be capable of removing odourous gases from the ventilation air of pig production buildings at low retention times.

No inhibitory effects should be observed when a biofilter is used to remove a cocktail of volatile fatty acids. Haner (1992) suggested the simultaneous utilisation of the carboxylic acids; this rate of utilisation was dependent on the molecular weight and the structure of each individual acid, with lower molecular weight un-branched acids being utilised most rapidly. This experiment verifies the effectiveness of biofilters treating exhaust ventilation air contaminated with volatile fatty acids.

Pressure drop increased for each stepped change in superficial gas velocities due to a greater flow resistance in the packing medium. For superficial gas velocities 38.2 m h⁻¹, 76.4 m h⁻¹, and 114.6 m h⁻¹ the corresponding pressure drops were 30, 78 and 120 Pa. No sudden change in pressure drop occurred across the biofilter for any of the volumetric loading rates. This demonstrated that the medium had good mechanical strength that led to negligible bed compaction and avoided short-circuiting during operation. Other researchers concluded that wood chip offers the most economically acceptable option as a biofilter medium with excellent stability properties even after wetting (Phillips et al., 1995). When compared to other packing

media such as compost, peat and coconut fibre, the pressure drop across wood chip is minimal and will reduce overall power consumption for operation of biofiltration systems (Sheridan et al., 2002b).

When bio-oxidising highly odorous compounds like *n*-butyric acid, near 100% removal efficiency is required to eliminate any odour impact. A geometric averaged odour detection threshold of $14.18 \mu\text{g } [n\text{-butyric acid}] \text{ m}^{-3} \text{ [air]}$ was obtained for *n*-butyric acid during this experiment. When *n*-butyric acid outlet concentration increased, the odour threshold concentration of the outlet air stream increased. At a volumetric loading rate of $143 \text{ m}^3 \text{ [air]} \text{ m}^{-3} \text{ [medium]} \text{ h}^{-1}$ and at a removal efficiency of 91.8%, the odour threshold concentration of the outlet air stream was $3920 \text{ Ou}_E \text{ m}^{-3}$. This odour threshold concentration could significantly increase the odour impact distance around a facility (Sheridan, 2002).

4. Conclusion

During the 120-day experiment, it was concluded that a moist enriched wood chip medium was an effective packing for the removal of *n*-butyric acid as long as nutrient mineral salts medium was applied to the biofilter. Upon investigation of the influence of superficial gas velocity on the degradation along the bed height, it was revealed that a smaller effective bed height than packed into the column (0.8 m) was required to eliminate the *n*-butyric acid in the inlet stream. The true elimination capacities (calculated for the bed height required) for *n*-butyric acid for superficial gas velocities of 38.2 m h^{-1} , 76.4 m h^{-1} , 114.6 m h^{-1} were 230 , 233 and $103 \text{ g } [n\text{- butyric acid}] \text{ m}^{-3} \text{ [air]} \text{ h}^{-1}$, respectively. This corresponded to inlet *n*-butyric acid concentrations of 3.1 , 1.19 and $0.24 \text{ g } [n\text{-butyric acid}] \text{ m}^{-3} \text{ [air]}$ for smaller effective true residence times of 30 , 11 and 5 seconds, respectively. The average pressure drops corresponding to these three superficial gas velocities were 30 , 78 and 120 Pa , respectively. A geometric averaged odour detection threshold of $14.18 \mu\text{g } [n\text{-butyric acid}] \text{ m}^{-3} \text{ [air]}$ was obtained during this experiment. It is important to note that

almost 100% removal efficiency is required due to this very low odour detection threshold for *n*-butyric acid.

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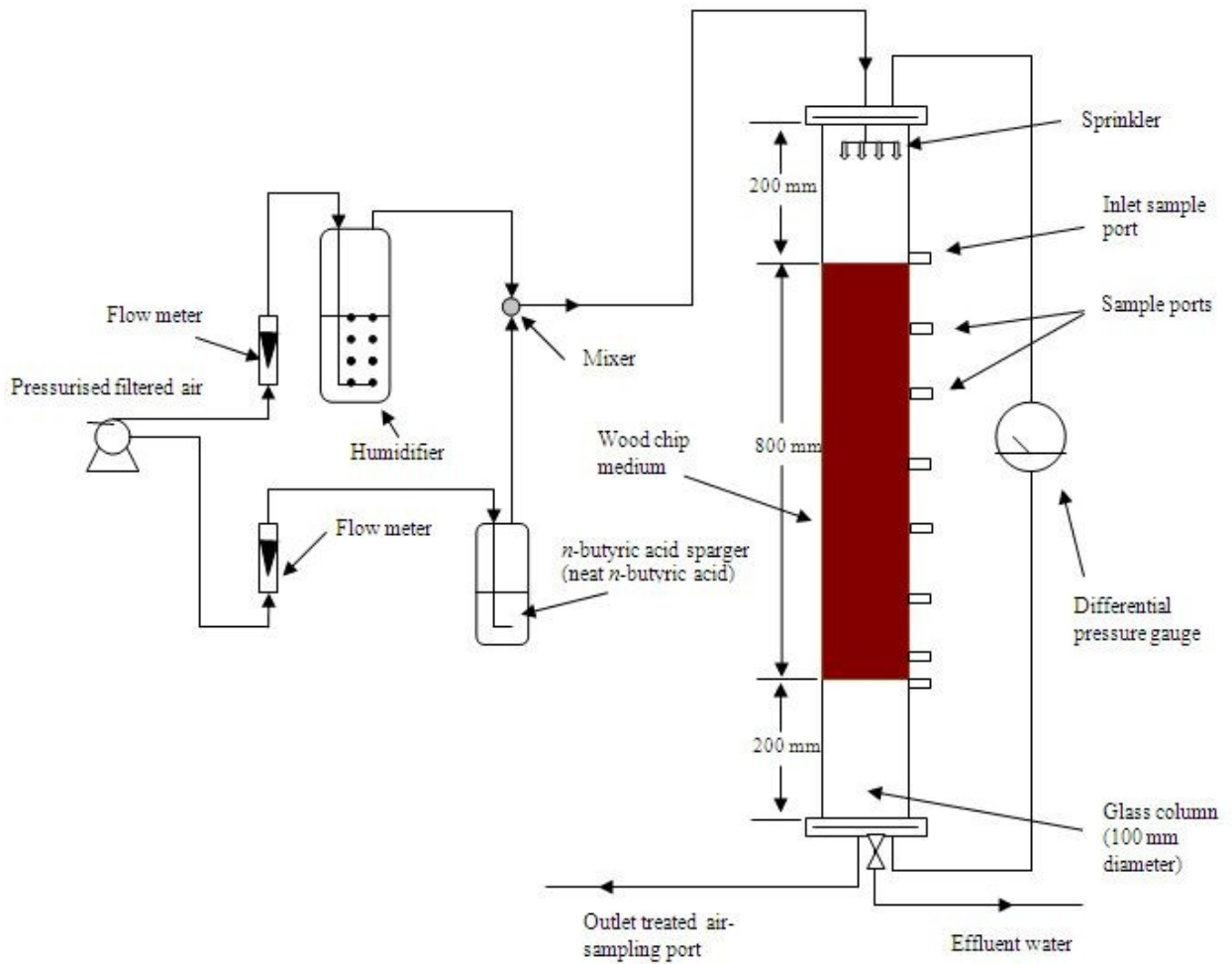


Fig. 1. Schematic diagram of biofiltration system