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UK Water Industry Research

COLLABORATIVE, IMPARTIAL RESEARCH

FATS, OILS AND GREASES (FOG) - WHERE WE ARE AND WHERE WE COULD BE

PROTOCOL FOR BIOLOGICAL DOSING INTO SEWER SYSTEMS

PART 1 - LABORATORY TESTING OF BIO-ADDITIVE DOSING COMPOUNDS

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PROTOCOL FOR BIOLOGICAL DOSING INTO SEWER SYSTEMS

1 Introduction

Sewer system operators estimate that FOG is a major factor in typically 30 to 50% of all sewer blockages. FOG is also a reason why pumping stations fail, in particular due to pump controls fouling and large blocks of FOG being ingested into pumps. All of these problems can lead to sewage flooding and environmental pollution incidents.

There are a number of ways that FOG deposition into sewer systems and pumping stations can be controlled, for example through better kitchen practice and the installation/ management of suitable grease interceptors at food service establishments. However, these measures are usually outside the direct control of Water and Sewerage Companies (WaSCs). For the foreseeable future it will be necessary for WaSC's to 'manage' FOG accumulations in the sewer network.

One approach to managing FOG accumulations is to dose the FOG deposits with microbial bio additives. These have been available for a number of years but, as of early 2015, had not been widely accepted as a viable approach by the majority of the UK Water Industry. One of the obstacles has been previous poor experiences with so-called FOG removal compounds. A decade ago a number of products of uncertain composition had been aggressively marketed as "biological treatment" and as a cure all for FOG problems in sewers. On many occasions these products were of little benefit or totally ineffective.

However, manufacturers and suppliers state that the products now on the market are a significant improvement over what was previously available. Most dosing compounds now digest FOG (total degradation to CO^2 and water) and don't simply break down the fatty acid bond. Experience gained with sewer system / pumping station dosing has provided practical experience and given a focus on the site related issues that need to be considered. Although, a small number of WaSCs are now routinely using dosing and finding that it is effective at the majority of locations, many sewer system operators are still reluctant to use biological dosing. There is, therefore, the potential for missed opportunities when, if applied correctly, bio additive dosing of the sewer system could be successful and help to reduce operational impacts and costs.

Another reason for WASC's being reluctant to use dosing has, until the publication of this Protocol in the summer of 2015, been a lack of recognised guidance. The reality has been that manufacturers often blame WaSC staff for a lack of awareness of how the products work and conversely WaSC staff in turn often suspect manufacturers are supplying ineffective products.

With the above in mind UKWIR funded a project to investigate the viability of FOG dosing into sewer systems. This project which was undertaken by WRc and Cranfield University commenced in April 2013 and was completed in April 2015. The objective was "*to produce*

standards that could be developed and applied to biological products, to give WaSCs confidence that they will be effective in the breakdown of FOG".

The research required the effectiveness of biological products used to treat / remove FOG from sewers / pumping stations to be investigated and to subsequently report on the issues that can impact on their effectiveness.

The tasks that were necessary to investigate the effectiveness of dosing programmes included:

a) Examining the factors that affect the potential effectiveness of bio additive products in treating and/or removing FOG from the sewers, pumping stations and treatment works inlets.

b) Considering the site conditions that could reduce (or negate) the effectiveness of bio additive dosing.

At the start of the UKWIR project, there was a general assumption that the success or otherwise of dosing lay primarily in the effectiveness of the dosing compound. However, after consulting with both those offering dosing services and WaSC staff that had significant experience of FOG related dosing in sewers/pumping stations, it soon became apparent that site related aspects were an equally / possibly more significant factor in the success of dosing programmes and these also needed to be considered.

This protocol therefore addresses both the compound effectiveness and site related aspects. This two part protocol gives details of:

- Part 1 - A laboratory based testing procedure, to establish if a particular compound has the ability to degrade FOG, through the observation of lipolytic activity.

- Part 2 - Site procedures and practices for undertaking the various stages of dosing in a sewer systems/pumping stations, from initial planning to routine maintenance dosing.

Provided that these procedures are followed it should enable dosing to be applied in a manner that is most likely to result in the required level of FOG reduction.

This short report outlines Part 1 of the Protocol. It explains the requirements and laboratory testing methodology to establish if a bio additive compound displays lipolitic activity.

2 Protocol Part 2 - Laboratory Testing Protocol

2.1 Need to assess the effectiveness of biological compounds

This laboratory based testing procedure is designed to assess if a particular bio additive product has the potential to be effective. This assessment is based upon tests designed to observe lipolytic activity.

However, it should be remembered that, even with a seemingly effective product the success or failure of a product will also depend upon the manner in which it is applied on site (see Part 2 of the Protocol – Catchment, site and deployment related aspects).

2.2 Development of a laboratory based test(s)

As part of this UKWIR research important new experimental work has taken place at Cranfield University to explore the practicalities of testing to assess the effectiveness of bio additives. This has demonstrated that testing requires many factors to be taken into account and failure to fully consider these would result in unreliable testing.

The development work established that it is necessary to consider each of the following aspects in some detail:

i) Specify a '*standard FOG laden effluent*'. This is necessary to provide a controlled source of nutrients and FOG, necessary for the bio additive to initially survive on and, in time, multiply.

ii) The effects of background bacteria present in the environment/water supply on the testing process.

iii) Investigate the importance of a range of parameters in sewer effluent. From this, select the parameters that are most likely to influence the effectiveness of a product and establish a broad operating range of bio additive compounds.

Parameters examined included effluent temperature, pH and dissolved oxygen levels and product contact time.

iv) Examine the effectiveness and reliability of extraction techniques, to identify an appropriate method to measure FOG removed from the test effluent.

From this work it became apparent that:

a) A '*standard FOG laden effluent*' could be developed and specified.

b) The water used to make up the effluent has to be autoclaved, to remove all background bacteria, which otherwise would have an uncertain effect on a controlled experimental process.

c) The parameters that have a significant effect on the ability of a dosing product to digest FOG are:

    i) Temperature and

    ii) Contact time

Nutrient levels and dissolved oxygen, provided there are adequate quantities present, have little influence on the performance of bio additives. The microorganisms have a high tolerance to variability of these parameters, provided that the nutrient and oxygen levels

are within the range typically experienced in sewers. (In practice a foul or combined sewer which is not anaerobic or septic should be suitable).

Similarly, pH has little effect provided it is above 5.

d) Extraction of remaining FOG from the standard test effluent is hindered by the levels of detergents interfering with the solvent extraction. Even when the procedures are undertaken with great care, there is the potential for a relatively wide spread of results.

Whilst it is possible to demonstrate lipolytic activity, it is not advisable to rank the effectiveness of different products against one another. This is because dosing compounds vary by design in terms of their microbial composition. As such, the different compounds become most effective at slightly different temperatures and after a different time interval. Likewise, no two sewers are the same and dosing will have a different impact, in terms of effectiveness and time taken to become effective.

Accordingly, a comparison of the performance of different compounds in a laboratory situation could give misleading information and, as such, is not recommended. Instead the laboratory based testing should primarily be used to establish if a compound demonstrates lipolytic activity and hence the ability to digest FOG. This is most important for compounds where there is no proven and documented track record of their successful use in FOG reduction dosing in sewers / pumping stations.

It is recommended that in the medium/long term laboratory testing results are backed up by a dossier detailing success rates in real life dosing in sewer systems and pumping stations.

3 Recommended Laboratory Testing Procedure

3.1 Introduction

The laboratory testing development work has enabled a procedure to be developed to assess the ability of a bio additive compound to degrade FOG, (i.e. through the observation of lipolytic activity). This procedure is detailed below. Bio additive products need to demonstrate lipolytic activity before they can be considered as potentially suitable for use in FOG dosing in sewers/pumping stations.

The laboratory testing and site investigations undertaken in an earlier stage of this research have highlighted that it is not possible to develop a testing programme that completely simulates sewer conditions, principally because sewer conditions are complex and will be unique for each location. Building up a dossier of a bio additives performance in real life dosing exercises will be necessary to provide such information.

The stages of the laboratory testing protocol are:

i) Preparation of the synthetic sewerage in which to undertake the tests.

ii) Addition of the bio-additives being tested.

iii) Batch testing.

iv) FOG extraction and calculation of the results.

3.2 Synthetic sewage preparation

Synthetic sewage should be used for the laboratory testing and not real sewage. This is because:

a) Real sewage is of variable consistency and, as such, cannot be used in controlled testing.

b) Health and safety considerations would make the testing more difficult to undertake.

Synthetic sewage is prepared using a sewage recipe modified from OECD guideline 302A & 303A (OECD, 2001). The solution consists of:

- Vegetable oil (1000 mg/L);
- Starch (200 mg/L);
- Meat extract (220 mg/L); (Sigma-Aldrich FLUKA 70164)
- Peptone from meat enzymatic digestion (320 mg/L); (Sigma-Aldrich FLUKA 70175)
- Urea (60 mg/L);
- Sodium chloride (56 mg/L);
- Anydrous di-potassium hydrogen (14 mg/L);
- Calcium chloride di-hydrate (8 mg/L); and
- Magnesium sulphate heptahydrate (4 mg/L) dissolved in tap water.

*Oil is added after autoclaving when using sterile conditions

Chemical Oxygen Demand (COD) of this high concentration synthetic sewage is measured using a colorimetric method (Spectroquant COD Cell tests (VWR UK). The COD should be approximately 1850 mg/L.

It is important that sterile conditions are used in the preparation process. This is because the testing undertaken during the development of this testing protocol showed that the use of non-sterile water resulted in the precipitation of FOG from the solution. The use of sterile conditions will limit the saponification process of free fatty acids formed by water endogenous microorganisms.

3.3 Bio-additives

Bio-additives are added to the synthetic sewage FOG mixture according to the bio additive manufacturers' instructions, including any pre preparation requirements.

3.4 Batch tests

All batch tests are conducted in triplicate.

Tests should be undertaken for a range of temperatures and durations. This is to assess the relative effectiveness of the bio additive at different temperatures and its effect over time. It is recommended that bio additives are tested under the following conditions:

- Approximately 17° C and repeated at 37° C.
- For durations of 10, 20 and 30 days.

Where a manufacturer/supplier makes a claim of effectiveness at lower temperatures, for example 5° C or 10° C, consideration should be given to undertaking an additional suite of tests at the lower temperature.

pH should be adjusted to 7.0 before the tests commence, using either HCl or NaOH.

The test should be run in aerobic conditions at the specified temperature (either 17° C or 37° C) and mixed using an orbital shaker for the specified duration (either 10, 20 or 30 days).

The flask size used on the orbital shaker is not crucial, provided that the ratio of flask size to liquid is 3:1 The rotation speed and rotation arm dimension of the orbital shaker is not critical, provided that the shaking motion is sufficient to keep the effluent/oil mixture in suspension, to prevent free oil from floating on the surface of the mixture.

Photograph 1 illustrates some of the samples prepared in the flasks.

Control samples are prepared in the same manner as the bio-added samples, the same volume of water should be added to control samples in place of the product to test.

Photograph 1 FOG/effluent samples in flasks during the testing

3.5 FOG extraction

FOG should be extracted from the samples following the period of incubation by following the standard operating procedure for solid phase extraction (SPE) method according to the EPA Method 1664 Revision A for Hexane Extractable Material (HEM) and modified by Cranfield University as follows.

1) Clogging issues due to high suspended solids (SS) are mitigated by employing a pre-filter comprising of a plug of glass wool inserted into the base of the reservoir.

2) All glassware should be prewashed in a laboratory dishwasher and rinsed with hexane to remove any residual oil and grease deposits.

3) A SPE disk and glass wool pre-filter should be inserted into the glass filtration apparatus with a Buchner flask employed for solvent collection. The apparatus and disc are washed by rinsing down the sides of the glassware with approximately 20 ml n-hexane dispensed from a wash bottle to remove any FOG contamination. The solvent should be drawn through the disk by vacuum pump and discarded appropriately. This step should be repeated and the disk allowed to dry under vacuum for two minutes.

   The disk should be conditioned by adding 10 ml methanol to the reservoir. A few drops of methanol are drawn through the disk, the system vented and the disc allowed to soak for one minute. The methanol should be drawn through the disk

leaving sufficient solvent to cover the surface and prevent the disk from drying before rinsing with 30 ml deionised (DI) water (and the waste discarded appropriately). The assembly is then transferred to a 500 ml Buchner flask.

4) The acidified sample (pH adjusted to <2) should be passed through the filtration apparatus under vacuum. The sample bottle should be thoroughly rinsed (by shaking) with approximately 50 ml DI water that is added to the reservoir just before the last of the sample passed through. The disk and the pre-filter are allowed to dry under vacuum for half an hour.

5) The filtration assembly should be transferred to a 100 ml vacuum flask (pre-rinsed with hexane) for FOG elution. The original sample bottle and cap are thoroughly rinsed by shaking with 10 ml hexane, which is then poured down the sides of the reservoir. A vacuum should be applied to draw a few drops of solvent through before allowing the disk to soak for two minutes, after which the remaining solvent is drawn through slowly. This should be repeated with a second hexane aliquot, with a third applied directly onto the glass wool pre-filter. Finally the sides of the reservoir are rinsed down with fresh hexane and the disk allowed to dry under vacuum for approximately five minutes.

6) The extract should be transferred to a 100 ml round-bottomed flask, which has been pre-rinsed with hexane, allowed to dry and weighed on an analytical balance. The residue should be rinsed twice with hexane and combined with the extract in the round-bottomed flask. The hexane is then evaporated using a rotary evaporator at 40°C and 100 mbar pressure. The flask allowed to dry at 40°C overnight or until a constant weight of HEM is achieved. FOG concentrations are calculated according to Equation 1.

$$FOG = \frac{m}{V}$$ Equation 1

Where m is mass of HEM extracted in mg, V is volume of sample in l, and FOG in mg/l.

Gravimetric analysis of FOG degradation

FOG removal values are calculated according to Equation 2**.**

$$FOG\ removal\ value = \frac{m_{original} - m_{residual}}{m_{orignal}} \times 100\%$$ Equation 2

Where $m_{original}$ is mass of originally added oil in mg, $m_{residual}$ is mass of HEM extract in mg.

3.6 Laboratory test pass fail criteria

The test results for different types of bioadditive should not be ranked to assess the most effective product. This is because products are not all designed for optimal operation at the same conditions so any ranking would not be comparing like with like. Also, products will require to be re-dosed at some stage, in general most frequently at the initial stages of a dosing exercise. This difference in dosing regimes would also make a like for like ranking problematic.

The main point of the laboratory testing is to demonstrate the presence (or not) of lipolytic activity from the bio-additive compound.

As a guideline, a product may be regarded as demonstrating lipolytic activity if the proportion of FOG removal increases with increasing contact time.

4 Discussion

A laboratory method for assessing the potential lipolytic activity has been developed.

This methodology can be used to demonstrate that, in laboratory controlled conditions, a compound demonstrates has the ability to degrade FOG depositions. This is particularly important for bio-additive compounds where there is as yet no track record of their effectiveness in the UK.

5 Recommendations

1) Manufacturers and suppliers of bio additive compounds have their compounds independently tested to demonstrate lipolitic activity and, as such, their ability to degrade FOG in sewers and pumping stations. Subject to successful testing being carried out, this will enable confidence to be gained that the bio-additive being considered has the potential to be effective.

2) It is not advisable to rank the effectiveness of different products against one another. This is because dosing compounds vary by design in terms of their microbial composition. As such, the different compounds become most effective at slightly different temperatures and after a different time interval.

   Furthermore, no two sewers are the same and dosing will have a different impact, in terms of effectiveness and time taken to become effective.

3) Bio additive dosing should be considered as an alternative to sewer cleaning as a FOG management methodology. This can often be a cost effective way of reducing FOG deposition related problems in sewers / pumping stations to a manageable level, with secondary benefits to the downstream network.