World Academy of Science, Engineering and Technology
International Journal of Chemical and Molecular Engineering
Vol:3, No:10, 2009

Effect of Anionic and Non-ionic Surfactants on Activated Sludge Oxygen Uptake Rate and Nitrification

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Abstract**—**A local wastewater treatment plant (WWTP) experiencing poor nitrification tracked down high level of surfactants in the plant's influent and effluent. The aims of this project were to assess the potential inhibitory effect of surfactants on activated sludge processes. The effect of the presence of TergitolNP-9, TrigetolNP-7, Trigetol15-S-9, dodecylbenzene sulphonate (SDBS) and sodium dodecyl sulfate (SDS) on activated sludge oxygen uptake rate (OUR) and nitrification were assessed.

The average concentration of non-ionic and anionic surfactants in the influent to the local WWTP were 7 and 8.7 mg/L, respectively. Removal of 67% to 90% of the non-ionic and 93-99% of the anionic surfactants tested were measured. All surfactants tested showed inhibitory effects both on OUR and nitrification. SDS incurred the lowest inhibition whereas SDBS and NP-9 caused severe inhibition to OUR and Nitrification. Activated sludge flocs sizes slightly decreased after 3 hours contact with the surfactant present in the test. The results obtained indicated that high concentrations of surfactants are likely to have an adverse effect on the performance of WWTPs utilizing activated sludge processes.

Keywords**—**surfactants, activated sludge oxygen uptake rate (OUR), nitrification, anionic surfactants, non-ionic surfactants

I. Introduction

SURFACTANTS are used in large quantities in domestic and commercial products, e.g. cleaning solutions, where they find their way to water bodies either through discharge of WWTPs' effluents or through infiltration where land application is employed as a method for disposal of effluent or of raw wastewater (e.g. onsite treatment of wastewater).

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Surfactants mainly consist of three classes: anionic, non-ionic and cationic. Anionic surfactants represent the major class of surfactants used in detergents and form about 41% of all consumed surfactants [1]. The predominant groups of anionic surfactants are linear alkylbenzene sulphonates (LAS) and linear alkyl sulphates (AS). Alkylphenol ethoxylates (APEs) are among the most widely used non-ionic surfactants. The most used commercial APEs products are Octylphenol ethoxylates (OPEs) and nonylphenol ethoxylates (NPEs). NPEs represent 80% of AEPs annual production. Many research studies reported that APEs degradation metabolites were more toxic than the parent APE surfactant and demonstrated endocrine disrupting characteristics [2]-[4].

Removal of NPEs in WWTPs was reported by many researchers. The authors of [5] reported that NPEs removal in WWTPs in the US was from 93% to 99%, but no distinction between cold and warm months was made. The highest level of NPEs detected in the influent was 33.7 mg/L whereas low levels as 0.005 to 0.26 mg/L were detected in the effluent. The authors of [6] monitored 9 WWTPs in different geographic locations in the US. The plants employed different types of biological treatment processes including activated sludge, trickling filter, oxidation ditch, lagoon and rotating biological contactor, and received ≤ 10% industrial wastewater. The authors reported that alcohol ethoxylates (AE) were effectively removed (>99%) in the activated sludge plants whereas plants with film biological processes (trickling filters and rotating biological contactors showed poor performance. T total AE concentrations in the effluents from the activated sludge and trickling filter type plants were around 0.92 and 15.6 µg/L, respectively. The total AE in the influent to the treatment plants reported in [6] ranged from 0.66 to 2.67 mg/L with an average of 1.53 mg/L. The activated sludge plants received the lowest concentration of 0.66 and 0.723 mg/L but the flow rates were not published therefore comparison based on loading was not possible. The authors of [7] investigated occurrence of different surfactants including nonylphenol, octylphenol and NPEs in 27 WWTPs in Japan and reported that NPEs removal in winter ranged from 66% to 99% which was generally lower than the 86% - 99% measured in autumn. Investigation of the performance of four WWTPs in Italy found that APEs removal ranged from 74% to 89% [8], [9]. The authors of [2] investigated the fate of nonylpehenolic surfactants in eleven WWTPs in the

area of Switzerland and Zurich for treatment plants receiving less than 10% industrial wastewater. They reported that the formation of biorefractory metabolites had a major impact on the removal of nonylphenol ethoxyaltes and found that temperature had a significant effect on the composition of the plant's effluent. According to [2] the concentration of NP in the influent to the eleven Swiss sewage treatment plants studied ranged from 1.12 to 2.06 mg/L and the overall removal of NP and metabolites compounds varied from 43% to 89%. The lowest rates and wide range of removal were attributed to the different processes and designs of the plants studied. Furthermore the authors reported a positive correlation between nitrification (measured as removal of ammonia) and removal of nonylphenol polyethoxylate oligomers (NPnEs) and individual NPnE, NP1E and NP which were classified with neglected formation under aerobic conditions.

Wastewater treatment plants (WWTPs) implement biological nitrification/ denitrification processes to achieve stringent nitrogen discharge targets. The nitrification process includes the conversion of ammonium-nitrogen to nitrite-nitrogen and nitrate-nitrogen by nitrifying autotrophic bacteria. Nitrifying micro-organisms are very sensitive to many factors, such as temperature, pH, and dissolved oxygen [10],[11]. In addition, the presence of toxicants in the wastewater treatment plant influent can inhibit autotrophic nitrifiers even at optimum nitrifying conditions.

Many research studies focused on the fate of surfactants in the environment but the effect of the presence of surfactants on the performance of activated sludge processes received little attention. The published literature to the knowledge of the authors of this article focused on the removal of surfactants in WWTPs or assessed potential toxicity using respiration tests. The literature concerning the toxicity of surfactants to activated sludge focused on the toxicity of specific surfactants to the respiration of activated sludge. The toxicity level of anionic surfactants reported spanned a wide range. The authors of [12], using respiration tests, found that LAS had no inhibitory effect on activated sludge, however using toxicity tests IC50 was measured at 120 mg/L.

A local wastewater treatment plant has been experiencing problems achieving nitrogen discharge limits, mainly due to poor nitrification, where either high ammonia concentration was detected in the effluent or, in many occasions, the concentration of nitrites was high. The problem was associated with poor settling in the secondary clarifier. Usually, to resolve this problem, the WWTP inoculate the aeration basin with commercial grown nitrifiers. The aims of this study were to assess the effect of increased concentration of both anionic and non-ionic surfactants present in the influent on activated sludge OUR and nitrification.

II. MATERIALS AND METHODS

A. Materials

Activated sludge samples were colleted from a local domestic wastewater treatment plant (WWTP). The samples were collected from the aeration basin early morning and the tests were performed on the same day of sample collection.

The anionic surfactants selected for this study were sodium dodecyl sulphate, SDS (Aldrich L-5750) and sodium dodecylbenzene sulphonic acid, SDBS (Aldrich D-2525). The non-ionic surfactants selected were of the nonylphenol ethoxylates group, Tergitol NP-9 Tergitol NP-7 and Trigotol 15-S-9 (Huntsman Chemicals). The stock solution of the surfactants were prepared with consideration to the purity of the surfactant and stored at 4 C.

B. Methods

Inhibition to activated sludge oxygen uptake rate (OUR) was carried out according to ISO8192 water quality – test for inhibition of oxygen consumption by activated sludge. Inhibition to nitrification was carried out according to ISO 9509 water quality – method for assessing the inhibition of nitrification of activated sludge micro-organisms by chemicals and wastewaters.

The local wastewater treatment plant was monitored over a week period both in May and June 2007. Twenty-four-hour samples were collected at three locations, influent, primary effluent and secondary effluent using auto samplers VST – 7750 (Manning Environmental Inc, USA), ISCO 3700 (John Morris Scientific Pty Ltd, AUS) and ISCO 2900 (Instrument specialties Co. Inc, USA) respectively. Each day a flow-weighted-composite sample was prepared from the twenty-four-hour samples and sent to an accredited commercial lab for analysis for anionic and nonionic surfactants.

C. Analytical Methods

The composite samples collected for monitoring purposes were analysed at an accredited commercial lab. The lab used the MBAS (methylene blue active substances) method number APHA 5540C (standard methods, 1992) and the KI-I2 method for anionic and non-ionic surfactants respectively. In the meantime duplicate samples were tested in the lab using the same analytical techniques.

The initial and final concentration of the anionic surfactant in the solution for the OUR tests was also determined according to MBAS method. Nitrite and nitrate concentrations were measured using HACH (DR400). Ammonium concentration was measured according to the Nessler method (standard methods, 1992), measurements were carried out at 420nm using a UNICAM UV/Vis Spectrophotometer.

III. RESULTS AND DISCUSSION

The first phase of this study involved monitoring of the local wastewater treatment under investigation at three locations on the treatment system, influent, primary clarifier effluent and secondary clarifier effluent for a week both in May and June 2007 during the periods 30/4-6/5 (days 1-7) and 5/6-10/6 (days 8-13). Daily composite samples were analysed for anionic and non-ionic surfactants. The concentration of anionic and nonionic surfactants in the influent and effluent are shown in Figures 1 and 2, respectively. The concentration of anionic surfactants in the influent ranged from 5.5 to 14 mg/L with an average of 8.7

mg/L. On the contrary the concentration of non-ionic surfactants showed a wide range varying from a minimum of 2 mg/L to 16 mg/L with an average of 7 mg/L. The highest non-ionic concentrations of 12 – 16 mg/L measured were for samples collected on 4 to 6 May, i.e. Friday to Sunday. Similarly the concentration of non-ionic surfactants on Saturday and Sunday, i.e. 9-10 June, were the highest among the data for that week. This trend was in agreement with the high concentrations of ammonia in the effluent and usually experienced on Monday of the week early autumn. Although the concentration of anionic was higher than non-ionic surfactants the removal of anionic surfactants ranged from 93.5% to 98.7% whereas the removal of non-ionic surfactants was generally low and varied from 42.9% to 90.0% (Table 1). The negative removal was due to an increase in the influent anionic concentration after the primary treatment, for example on day 2 (i.e. 1/5/2007) the influent and primary effluent concentrations were 4 and 8 mg/L, respectively.

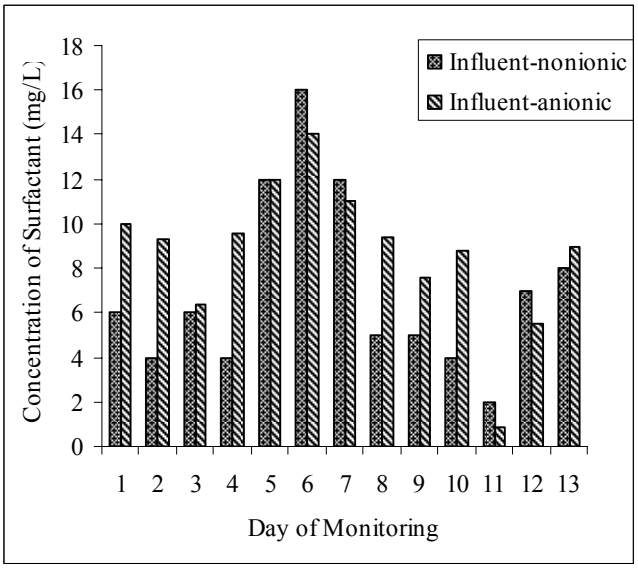


Fig. 1 Concentration of anionic and non-ionic surfactants in the influent to a local WWTP

Although a similar trend was observed for anionic surfactants the high removal offset the increase in concentration measured after primary treatment. No further work was carried out to identify the cause of increase both in anionic and non-ionic concentration in the primary effluent measured on 1/5 – 4/5, 7/6 and 9/6. It was also observed that the treatment plant performance in May was poor especially for nonionic surfactants removal, compared with that in June. The concentrations of surfactants in the influent measured at the local WWTP were higher than those published in the literature for WWTPs in US [6], Italy [8], [9]. Japan [7] and Switzerland [2] where the highest nonionic concentrations were around 2 mg/L. However, a similar range of concentration was reported in [5] for NPEs concentrations in influent to WWTPs in the US.

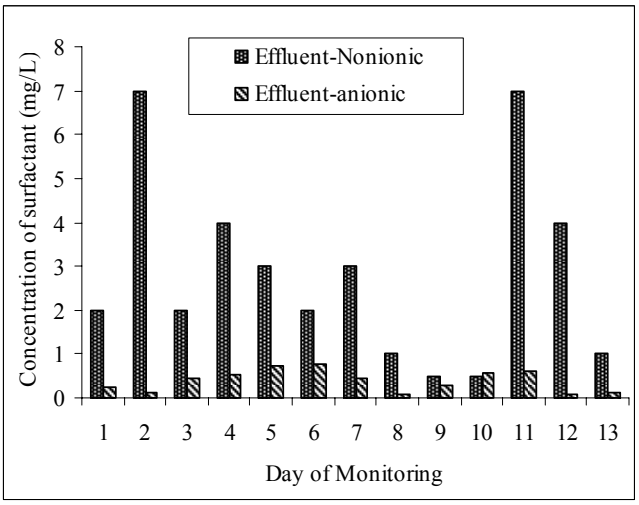


Fig. 2 Concentration of anionic and non-ionic surfactants in the effluent from a local WWTP

TABLE I
REMOVAL OF ANIONIC AND NONIONIC SURFACTANTS AT A LOCAL WWTP

Date	Day No.	anionic surfactant	nonionic surfactant
30/04	1	97.6%	66.7%
1/05	2	98.7%	-75.0%
2/05	3	92.8%	66.7%
3/05	4	94.7%	0.0%
4/05	5	94.0%	75.0%
5/05	6	94.5%	87.5%
6/05	7	96.0%	75.0%
5/06	8	98.9%	80.0%
6/06	9	96.4%	90.0%
7/06	10	93.5%	87.5%
8/06	11	27.1%	-250.0%
9/06	12	98.4%	42.9%
10/06	13	98.6%	87.5%

Potential inhibition of surfactants to activated sludge was assessed both in terms of inhibition to OUR and nitrification. Inhibition to OUR for a given concentration of the selected surfactant was measured as the reduction in the activated sludge oxygen uptake in the presence of the surfactant relative to that in the absence of the surfactant. The effect of the presence of each of the selected surfactants SDS, SDBS, Trigotol NP-9, Trigetol NP-7 and Trigetol was assessed for concentrations of 1 to 100 mg/L. OUR tests carried out in the presence of SDS showed an inhibitory effect which increased from 12.9% to 44.2% for SDS concentrations of 10 to 100 mg/L. Similarly, SDBS showed an inhibitory effect proportional to the initial concentration. However SDBS inhibition was more severe than that incurred by SDS, ranging from 27.6% to 75.5% for 10 to 100 mg/L. These results indicate SDS is more biodegradable than SDBS, which could

be attributed to the presence of benzene and its effect on the mechanism of biodegradation of SDBS. According to [14],[15] the mechanism of the breakdown of LAS involves degradation of the straight alkyl chain, the sulphonate group and finally the benzene ring. They explained that breakdown of the branched alkyl group is more complex than straight chain where degradation can not be through oxidation by microorganisms rather it must be through loss of carbon atoms one at a time.

Inhibition to activated sludge OUR in the presence of Trigetol NP-9 and Trigetol 15-S-9 is shown in Figure 3. Trigetol inhibition ranged from 27% at 1 mg/L to almost 60% at 100 mg/L. Trigetol 15-S-9 inhibition to OUR was around 20% lower than that measured for Trigetol NP-9 at all concentrations tested. A similar trend was also observed in the presence of Trigetol NP-7.

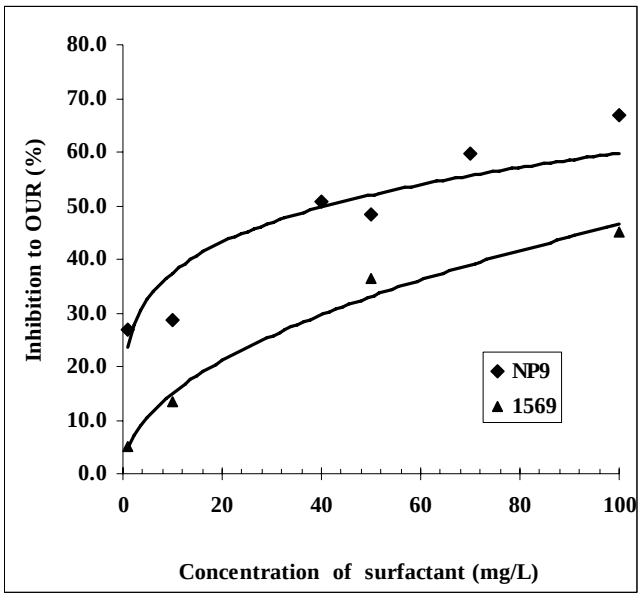


Fig. 3 Inhibition to activated sludge OUR in the presence of the nonionic surfactants Trigetol NP-9 and Trigetol 15-S-9 for concentration from 1 to 100 mg/L

The data shown in Figure 3 are average of data collected from at least triplicates OUR tests using activated sludge samples collected August 2007. All OUR tests were carried out using activated sludge samples collected on the day of the test. It was noticed that inhibition extent and trend varied with tests performed in May showing higher inhibition and more sensitivity to the presence of the surfactants compared with inhibition using samples colleted July-August. The inhibition to OUR measured in the presence of Trigetol NP-9 obtained using samples collected in May were in the range 70-80% both for 1 and 10 mg/L and around 100% for 100 mg/L. Samples collected in July showed lower inhibition for the low concentrations tested, 1 and 10 mg/L but inhibition at 100 mg/L increased with increased exposure time reaching 100% after 100 min of contact (Figure 4). The increase inhibition with time observed for 100 mg/L indicated that the activated sludge microorganisms were more acclimatized to surfactants compared with those collected in May. A similar trend of varied extent of inhibition was observed for Trigetol NP-7 (Figure 5A and B). Inhibition using activated sludge samples collected in May (Figure 5A) increased at a high rate during the 2h test using samples collected in May but a lower rate if level of inhibition was measured using samples collected in August (Figure 5B). In addition the trend shown in Figure 5B, i.e. little variation in inhibition was measured after about 60 min of exposure to the surfactant which indicated that the activated sludge population in this sample was more acclimatized to surfactants.

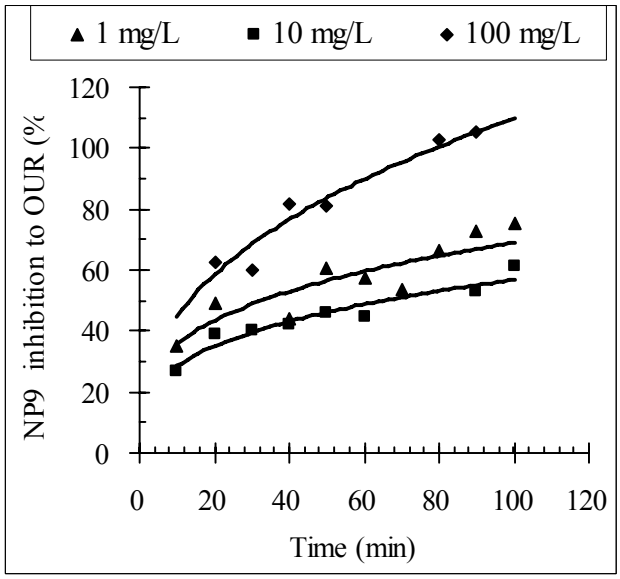
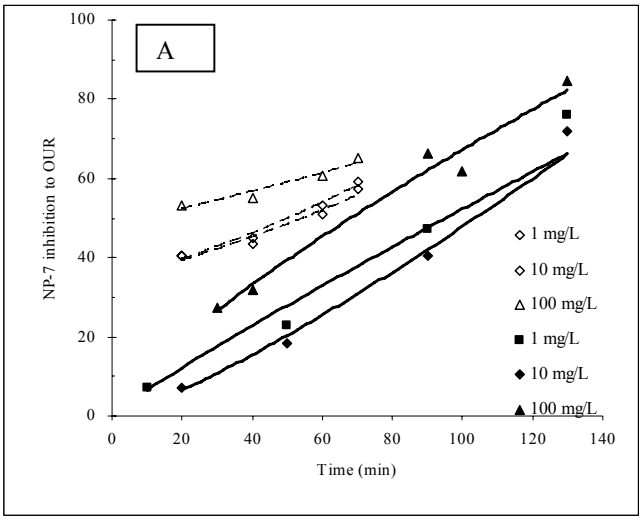


Fig. 4 Inhibition to OUR for different concentrations of Trigetol NP-9 during a 2h OUR test using activated sludge samples collected July 2007

The effect of surfactants on activated sludge flocs was also assessed using microscopic images of activated sludge flocs collected from the reactors used for OUR tests. Results obtained for flocs exposed to SDS and SDBS are included in this article. Two of the images obtained in the presence of SDBS are shown in Figure 6. Three samples were collected at the end of each OUR test, then for each sample 5 – 10 images were captured and analysed for flocs mean projected area and perimeter. Analysis of data obtained showed that the mean projected area and the perimeter of the flocs decreased with increased concentration of the surfactant both for SDS (results not shown) and SDBS compared with those for the control reactor (i.e. in the absence of the surfactant) for all concentrations tested. These results suggest that changes to the characteristics of the flocs may lead to poor settling behaviour in secondary clarifiers which also suggest that surfactants in the influent may be a major factor of the washout usually experienced early autumn in the local WWTP.



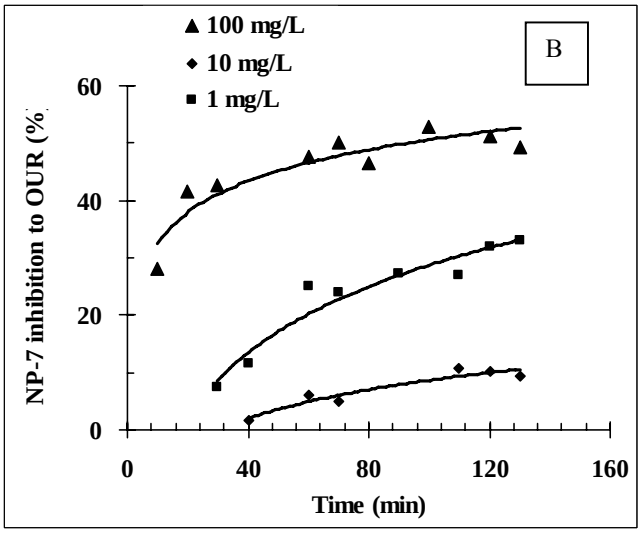
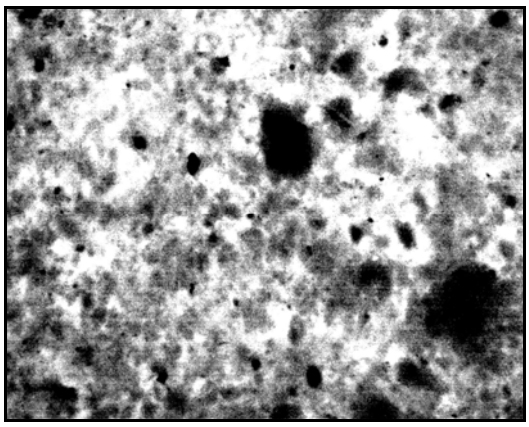
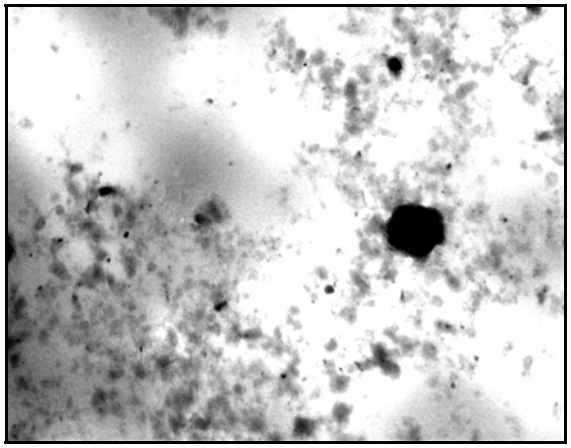


Fig. 5 Inhibition to OUR for difference concentrations of Trigetol NP-7 during a 2 hr test using activated sludge samples collected in May (A) and samples collected in August (B)



(A)



(B)

Fig. 6 Microscopic images of activated sludge flocs at the end of OUR tests for SDBS concentrations at 10°C (a) 10 mg/L (b) 100mg/L SDBS (images at magnification 100×with blue filter)

To asses whether the inhibition effect observed for SDS and SDBS was peculiar to the treatment under investigation (referred to as AS#1) OUR and nitrification inhibition testes were carried out using activated sludge from a different WWTP (referred to as AS#2) receiving similar influent, i.e. mainly domestic wastewater. Although SDS inhibition to AS#2 and AS# were almost of the same magnitude (figure not shown), SDBS inhibition to AS#2 was 5% – 20% lower than that observed for As#1 (Figure 7). The different level of inhibition for the two sludge samples could be attributed to the types of micro-organisms in each activated sludge sample and indicate that AS#2 sludge was more acclimatized to anionic surfactants. In future research potential relationship between the activated sludge micro-organisms in the samples and inhibition values will be investigated.

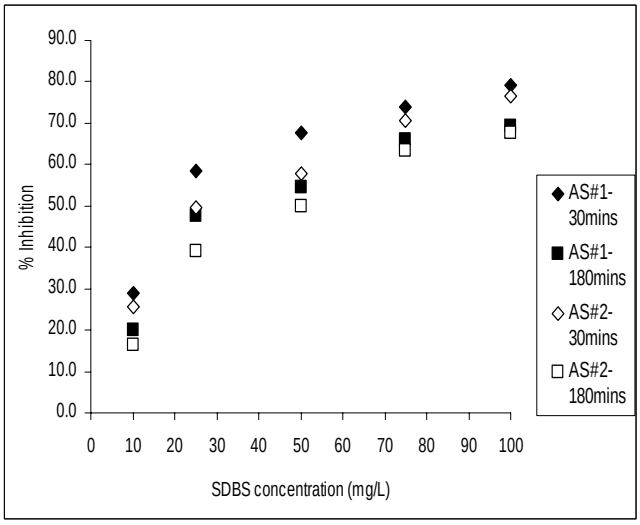


Fig. 7 Inhiobition to OUR in the prersence of SDBS

Inhibition to nitrification was measured in terms of the reduction in the production of oxidised nitrogen (i.e. ammonia

oxidation to nitrite and nitrates) compared with that in the absence of the surfactant. Inhibition to nitrification in the presence of SDS is shown in Figure 8. The results showed that inhibition to nitrification in the presence of SDS was proportional to SDS initial concentration. The level of inhibition calculated in terms of drop in oxidised nitrogen production was lower than the level of inhibition calculated in terms of reduction in ammonia removal. This could be attributed to the utilisation of ammonia for activated sludge microorganisms' growth. The trend observed in Fig. 8 suggests that for SDS concentrations less than 50 mg/L the effect on the nitrifiers in the activated sludge sample, consequently the drop in the rate of nitrification, was more than the drop in the rate of ammonia utilisation by the activated sludge microorganisms for growth (cells synthesis). Inhibition to nitrification shown in Fig. 8 was in agreement with the results obtained for SDS inhibition to OUR. The level of inhibition to OUR for SDS reached about 17.4% at 50 mg/L and 27.4% at 100 mg/L SDS (after 180 minutes) which indicate that the inhibitory effect of SDS were more pronounced for SDS concentrations less than 50 mg/L. SDBS inhibition to nitrification (results not shown followed a similar trend compared with that for SDS, but the level of inhibition measured for SDBS was higher than that measured in the presence of SDS. This could be contributed to the lower biodegradability of SDBS due to its structure and the presence of benzene. The IC20 (20% inhibition) for SDBS was 11.95 mg/L compared with 21.30 mg/L for SDS. Similarly IC50 was 74.4 mg/L for SDBS compared with 193.6 mg/L SDS

Inhibitions to nitrification in the presence of TrigetolNP-9, Trigetol NP-7 and Trigetol 15-S-9 are shown in Figures 9-11 respectively. The level of inhibition to nitrification incurred by the presence of 1 – 100 mg/L Trigetol NP-9 showed a sharp sigmoidal shape, an inhibition of 26-33% was measured for concentrations of 1 – 25 mg/L. A sharp increase in inhibition was observed at concentrations higher than 25 mg/L reaching 70% at 40 mg/L Trigetol NP-9 and reached a plateau at 100% for concentrations higher than 50 mg/L.

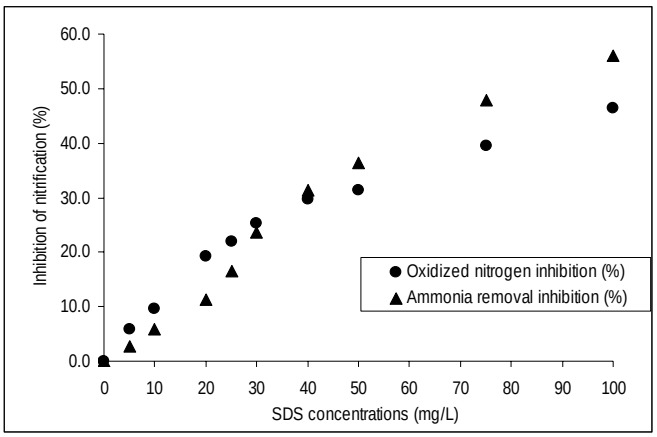


Fig. 8 Inhibition to Nitrification in the Presence of SDS

Tests performed using Trigetol NP-7 and Trigetol 15-S-9 also showed a rapid increase in inhibition to nitrification with increased initial concentration from 1 to 30 mg/L at which an inhibition of 50% and 45% was measured for NP-7 and 15-S-9, respectively. Inhibition for the higher concentrations tested up to 100 mg/L increased to 60% for both surfactants. The results shown in Figures 9-11 indicate that Trigetol NP-9, NP-7 and 15-S-9 have inhibitory effects on nitrification with NP-9 having the most severe inhibition to activated sludge nitrification compared with other surfactants tested.

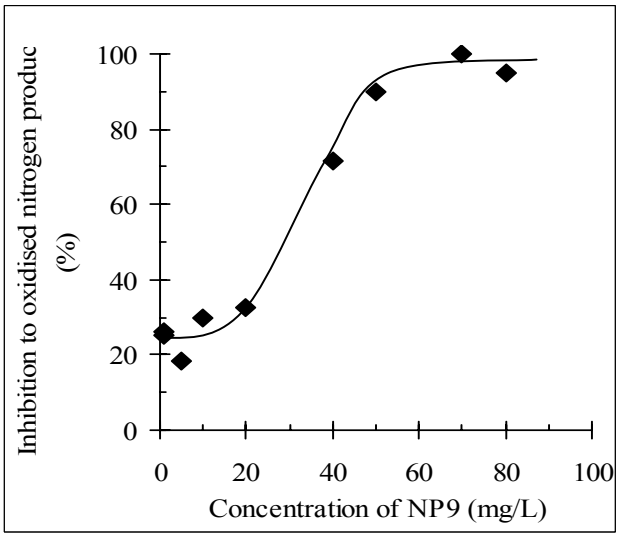


Fig. 9 Inhibition to oxidized nitrogen production in the presence of the non-ionic surfactant Trigetol NP-9

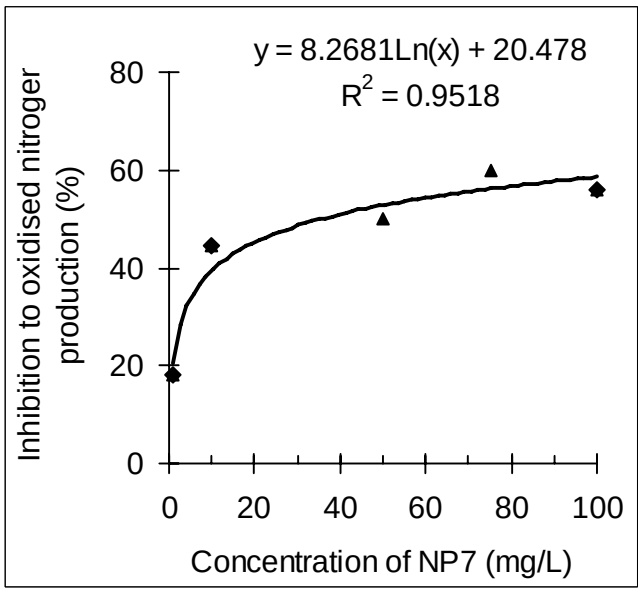


Fig. 10 Inhibition to oxidized nitrogen production in the presence of the nonionic surfactant NP7

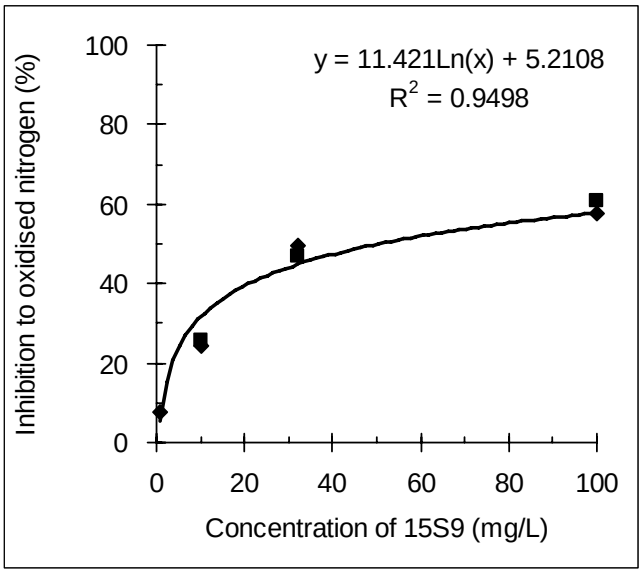


Fig. 11 Inhibition to oxidized nitrogen production in the presence of the nonionic surfactant 15S9

IV. Conclusions

The anionic surfactants SDS, SDBS and the non-ionic surfactants Trigetol NP-9, Trigetol NP-7 and Trigetol 15-S-9 had inhibitory effects on activated sludge OUR and nitrification. SDBS and Trigetol NP-9 showed severe inhibitory effects of up to 100% inhibition to OUR compared with that in the absence of the surfactant for concentrations higher than 30 mg/L.

The morphological images and parameters of activated sludge flocs showed that SDS and SDBS can have significant adverse effects on activated sludge flocs measured in terms of mean projected area, perimeters and equivalent diameter. Overall, the presence of both SDS and SDBS resulted in reduction in the sludge flocs size, which means that the presence of SDS and SDBS may lead to poor solids settling in the secondary clarifier.

The tests carried out to measure inhibition to OUR although was reproducible for the same activated sludge sample varied by 20-30% in some cases and showed different trends with respect to inhibition versus time. This effect was tracked down to be linked to date of collection of the activated sludge sample which indicate that the activated sludge population and operating conditions in the plant play a major role in controlling the response of activated sludge micro-organisms to inhibiting surfactants. Further research is in progress to assess effects of surfactants under continuous flow conditions on activated sludge OUR, nitrification and process performance.

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