A NEW AND NOVEL ABIOTIC-BIOTIC FOULING SENSOR FOR AQUEOUS SYSTEMS

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ABSTRACT

Deposits on surfaces in water – bearing systems, also known as "fouling", can lead to substantial losses in the performance of industrial processes as well as a decrease in product quality and asset life. Early detection and reduction of such deposits can, to a considerable extent, avoid such losses. However, most of the surfaces that become fouled, for example, in process water transport pipes, membrane systems, power plants, food and beverage industries to name a few, are difficult to access and the analysis of the water phase do not reveal the extent of the deposits. Furthermore, it is of interest to distinguish between biological and nonbiological deposits. Although they occur together, different counter measures are necessary. Therefore, sensors are required that indicate the development of surface fouling in real time, non-destructively, in-situ and can discriminate between abiotic and biotic based deposits. A new and novel sensor has been developed that provides said discriminate detection by utilizing conventional heat transfer reduction sensory coupled with ultrasonic detection of materials on the same surface concurrently. The technical aspects of the design, operation, and application will be discussed in the paper. Real time graphical detection followed by automated reduction control runs will also be presented as well as revealing if the deposit is biotic or abiotic.

INTRODUCTION

One of the main causes of performance loss, quality and runnability problems in industrial systems is related to contaminants and deposits. These deposits are composed of inorganic, organic or microbial matter, respectively. Most of the deposits contain various or even all types of these contaminants and form complex matrices. Of these, microbiological contaminations, also named biofouling, are one of the biggest issues and risks in water bearing industrial systems. They cannot only cause deposits that impact the function and efficiency of the systems. They often are the cause for health risks (e.g., Legionella). Fouling can be generalized into 4 forms, inorganic, suspended solids, organic, and microbiological. Of these forms of fouling, it is only inorganic crystallization fouling that does not lead to the worst form of corrosion, namely localized. This type of corrosion eventually transitions into high pitting penetration rates that drastically reduce the asset life.

The majority of the fouling which occurs in aqueous systems are detected indirectly by means of reduced process side throughput, increased time to get to operating temperature and or pressure, pressure drop, approach temperature increase, or the use of extensive instrumentation to calculate "at that time" heat exchange U coefficients and or cleanliness factors. Under certain circumstances, some of these methods are not sufficiently accurate unless normalized. Or the measurements taken have not been corrected for cooling water or process flow changes, shear stress change and bulk cooling water change or surface temperature change. There may be a large lag time to foulant detection which can lead to foulant aging and dehydration to a point of being irreversible fouled, where-by chemistry and chemical adjustment in the water side environment would not provide cleansing of the surface and maintain a clean state. An example would be the comparative time for a side stream annular heat transfer test section to detect fouling of a well instrumented utility surface condenser, where in they were both operated at the same surface temperature and shear stress (velocity corrected for the geometry) on the same cooling water (Beardwood, 2011). The steam surface condenser heat transfer surface area for 175 MW would be 150,000 ft² (13,935 X10⁶ mm²) would require a large quantity of foulant coverage to be detected compared to the annular test section which has 0.05 ft² (4645 mm²) of foulant detection surface.

CONCEPT DEVELOPMENT AND LABORATORY WORK

Although methods had been developed to detect and identify the four forms of fouling and corrosion utilizing a grouping of on-line analyzers in conjunction with the annular heat transfer test section (Beardwood, 1996, 2000, 2003, 2011, 2016), a desire for more specificity from a singular analyzer was desired. An interest in ultrasonic measurements of deposit thickness began in late 2006, with a prototype being developed by 2008 which could detect down to 0.001 mm (1µm) thickness (Seida, Flocken, Pöschmann, Schultz, Klaua and Dietrich, 2009). By using a specific ultrasound frequency between 5 and 15 MHz at a speed of 1480 m/second @ 20°C, the lag time between transmission and reflection can be measure with a resolution of 120 picoseconds (< 1 nanosecond). Thickness determination was based upon time of flight of the sound between transmission and reflection. The transducer emits an ultrasonic signal to the surface to be measured (either the reflector plate or a pipe wall) at a predefined interval and then receives the reflected echo. The time interval between the emitted signal and its echo is used to calculate the distance between the sensor and reflector surfaces. If scale forms on the surface of the reflector, then the time required for the ultrasound wave to complete its circuit is reduced. This result is measured as the difference in value in relation to the "zero point" set at the beginning of a measuring cycle. Figure 1 depicts the change in time for the ultrasound signal to pulse and echo on a clean versus a scaled surface. The time data are converted to scale build-up or removal, as the time either decreases or increases, respectively. This allows the analyzer to continuously monitor the thickness of any build-up on the target surface, with positive values indicating a scale forming environment and negative values showing the removal of scale. The accuracy of measurement and resolution of the value is high enough (1 um) to be applicable for monitoring and controlling the effectiveness of a scale control chemical program in real time. The sensor is illustrated in Figure 2 and results of a field application is provided in Figure 3. A number of challenges needed to be overcome in order to have accurate results. These were completed prior to 2010. Onboard algorithms were developed to correct/compensate for changes in bulk fluid temperature, pressure and ionic strength; which affect the speed of sound. An example of the influence of temperature and conductivity on the sound velocity is illustrated in Figure 4. For example at 20°C the error in thickness calculations is about 30 µm per 1°C and 7 µm per 1 mS of conductivity at 10 mS conductivity water.



Fig. 1 Ultrasound impulse and reflection for a clean and scaled surface, with a lower time value (t_s) for a scaled surface.



Fig. 2 Analyzer probe showing the transmitter and removable reflector plate.



Fig. 3 Analyzer output showing the effect of an antiscalant on reducing the rate of scale build-up, and removing scale (negative rate of build-up) if dosed at a sufficient level.



Fig. 4 Sound Velocity Dependency of Temperature and Conductivity in Water.

Self-Scaling Monitor

These sensors are used in fluids that are "self-scaling" or fouling; that is, the system has suspended solids or the scalant concentration has exceeded the solubility limit in the bulk fluid. Also known as a homogenous exsitu crystallization process in which the fouling is mass transfer dominant. It was further postulated that heat exchanger fouling could be simulated provided the sensor reflector cap was heated. This was found to be misleading as the results only validated that much of the deposition was on the backside from reverse eddies as the water past the probe. Heat exchanger fouling that are not associated with suspended solids are insitu, attachment dominate and scales are from heterogeneous crystallization. Additional information regarding the self-scaling monitor can be found in the associated patent (Seida et al., 2009).

Heated-Scaling Monitor

Obviously the corrective actions in the self-scaling unit could be transferred to a heated unit, however there were many technical/scientific design hurdles that needed to be addressed in order to generate data and graphical trends that were informative, aligned to known real world outcomes and in real time. The individual items that were required to be engineered and designed in order to achieve a functional heat transfer test section device in combination with thickness measurements were as follows:

- A sample conditioning conduit with flow normalization and test section placement determination.
- Equivalent shear stress development from flow velocity for the geometry compared to the known calculated shear stress simulation values of the annular heat transfer test section as associated to other heat exchanger cooling water channel geometries (Beardwood, 2011).
- Constant flow maintenance.

- Design and construction of the ultrasonic sensor probe and associated heated target surface test section. Polyether ether ketone (PEEK) insulation and thermal conduction material of construction required the use of a Copper Nickel Alloy.
- Design and mounting of the heat transfer test section, over 7 iterations.
- Heater cartridge sizing for specified bulk water, and heater surface temperatures and flow conditions, and a supply of constant clean electrical power (CFD modelling).
- Core material selection to achieve the upper limit surface temperature required and repeatable thermal couple placements (T2; near the wetted end surface and T3; near the heater cartridge) for calculating fouling factors.
- Bulk water temperature correction (T1) for calculating fouling factors and fluid density and kinematic viscosity of the water. Without these corrections the calculated fouling factors would contain a false positive (i.e., more) or a negative (i.e., less) component.

The final design sample conditioning conduit and scale thickness and heat transfer reduction detectors is illustrated in Figure 5 (Seida, Flocken, Bierganns, and Schultz, 2013). More internal illustrations and the conventional fouling factor calculations are outlined in the "OnGuardTM 3H Analyzer, Scaling Control, Manual Version 1.100 EN, 06-FEB-2017" and associated patent literature (Bliss & Patterson 2014).



Fig. 5 Side Stream Sample Conditioning and Sensory Detection Hardware.

Laboratory runs (Bliss & Patterson, 2012) with the final heated scale monitor design (OnGuardTM 3H analyzer; 96 Watts of electrical power) against the annular scale monitor (OnGuardTM 2-plus analyzer; 1500 Watts of electrical power) with primarily a calcium phosphate scale formation are illustrated Figure 6 and Figure 7 under equivalent shear stress and surface temperatures. The conditions for the 2 tests were; bulk water temperature of 24°F, annular monitor flow of 26.3 l/min (1.8m/sec; Rn = 14,850), new scale monitor flow of 18.1 l/min (0.75 m/sec; Rn = 14,850), surface temperatures of 60°C (Test 1) and 67°C (Test2). The fouling factor is in units of 10⁻⁵ X °F ft² hr / Btu. It can be seen in Figure 6, that the 2 monitors track quite well for fouling factors and thickness and that the relationship between thickness and fouling factors are very close (i.e., 50 and 100 μm for 25 and 50 Fouling Factors; 2 μm per Fouling Factor). The later relationship is born out in Figure 7 results for the linear relationship; $R^2 = 0.9979$. Another laboratory (Bierganns, 2013) measured the deposit thickness mechanically with a micrometer and obtained equivalent results compared to the ultrasonic detected values of 35, 90, 130 and 150 μ m, with calculation accuracy down to 5 μ m. The associated Reynolds Number for the 2 monitor's sample conditioning conduit past the heat transfer test sections are illustrated in Figure 8.

A laboratory run was conducted to evaluate the ability to respond to deposit thickness increase. Figure 9 illustrates the test outcome. A reduction in the antiscalent chemical feed pump occurred until the set point was exceeded, next a booster chemical feed pump is activated to quickly return the antiscalent concentration. The feed optimization starts over again reducing the antiscalent feed/concentration until it once again exceeds the set point. Overall, the newer fouling monitor was found to operate at lower flow rates and about 1/20th the power than the annular monitor and lower target test sizing at 396 mm²; with equivalent results without conflicting operational variables. That is; compared to the standard, all the hurdles had been overcome. Additional information regarding the design of the heated scaling monitor can be found in the associated patent literature (Bliss and Patterson, 2014, Seida et al., 2013 and 2015).



Fig. 6 Annular vs New Heated Scale Monitor Deposit Fouling Factors and Thickness.



Fig. 7 New Heated Scale Monitor Calculated Fouling Factor vs Thickness.



Fig. 8 Comparison of the 2 Monitor's Reynolds Number vs Flow Rates.



Fig. 9 Response to Scale Thickness Increase; Laboratory Run.

Heated-Bio-Fouling Monitor

During the research and development associated with the heated scale monitor, additional studies were performed concurrently to determine if biofilms could be detected. It is well known that heated surfaces colonize faster with higher biovolumes, higher percent moisture, and higher ratios of anaerobic to aerobic bacteria in the wild (Beardwood & Therrien, 1999). The main issue was, could biofilms be seen on a substrate surface in water, as their acoustical impedance were very similar. Initially, soft organic films of oil, grease and gelatin were trialed as a biofilm surrogate and then insitu biofilms. Modifications of the ultrasonic settings were performed to measure at various degrees of sensitivity and at high sensitivity there was high noise and secondary sound scatter in the liquid (i.e., high noise distortion at higher signal to noise ratios) when ultrasound amplification was performed. Upon changing the design and set-up of the ultrasonic probe, these issues were resolved. The work revealed that at various sensitivity levels (i.e., low, medium and high) deposition could be detected. Hard inorganic scale responded at lower sensitivity level compared to organic materials or even biofilms that only responded to the highest sensitivity settings. One reason for this is the different values of ultrasound reflection as illustrated in Table 1. It was also noted that the corresponding reflective sound signals could not only be used for measuring but also to differentiate the type of deposit. Hence the process could be tailored to determine all three forms of deposits (Seida et al., 2015; patents WO 2013/092819 A3 and US 2015/0000406 A1)

Table 1.	Percentage	Distribution	of Ultrasou	nd Reflection.
	0			

Material	Acoustical	% Deposit	% Surface
	Impedance		Substrate
	10^6 kg/m ² sec		
Water	1.5	-	~ 100
Biofilm	1.7	0.4 (soft	86.0 (hard
		surface, weak	surface,
		signal)	strong signal)
Scale	20	74.0 (hard	0.5 (hard
		surface,	surface, weak
		strong signal)	signal)
Steel	45	~ 100	-

Outside laboratory work was contracted to test the accuracy of the biofilm monitor in calculating biofilm thickness (Wagner 2016, 2017). The laboratory utilized Optical Coherence Tomography (OCT), a state of the art technology in academia to perform biofilm thickness measurements non-destructively and in-situ (Wagner 2016 and 2017). The results revealed a good coefficient of correlation; $R^2 = 0.9094$, for thicknesses in the range of 25 to 100 µm. A result is illustrated in Figure 10, where-in the thickness reading on day 1 resulted in a measurement of 27 μ m from the OCT (+/- 11 μ m) while 26 μ m with the ultrasound measurement at the same point of time. On day 3 the OnGuard[™] 3B analyzer measured 100 µm while the OCT vielded 86 µm (+/- 33 µm). The differences in OCT vs ultrasound is the area of observation, where-in the biofilm architecture changes due to degree of cross-linkage and presence of incomplete and uneven surface coverage. Below

10 μ m deposit thickness was difficult to see and calculate values, while the OCT technique could. It was found that this could be overcome by using a temperature calculation as the leading indicator of biofouling initiation. A value of 0.2°C Delta Temp ISO is where the biofilm as a structure begins to form. It can be seen in Figure 11 that the thickness begins to be detected at 0.25°C and trends well with the Delta Temp ISO. Delta Temp ISO is defined as the difference between the calculated heated surface temperature (Ts) and the bulk water temperature (T1). While the surface temperature of the heated target can be calculated by the following:

 $T_s = T_2 - (T_3 - T_2) X RTD$ Distance Ratio ([RTD₂ distance to heater surface / RTD₃ distance to RTD₂] = 0.71)



Fig. 10 Heated Bio-Fouling Monitor Thickness Reading vs OCT Determination.





The Delta Temp ISO and biofilm thickness relationship can also be seen in Figure 12, where-in the time calculated from the graph to get attachment to the surface (i.e., Delta Temp ISO positive values prior to thickness detection and trending increase at 0.2°C) was found to be 2.84 days which very reasonable and in line from what was determined utilizing an annular test section in the field (Beardwood, 1995, 1996). It was unknown if the data to the left with positive and negative temperature values represent an attachment / detachment processes (Epstein, 1997) and the time to colonize the surface here, maybe a Quorum Sensing period. The laboratory data was rechecked and it was found that all variables (i.e., flow, bulk water temperature, Delta Temperature ISO, power, and conductivity) were constant without variational swings. The delay period of variable Delta Temperature ISO was an attachment / detachment process (Epstein, 1997). It had been seen in other inorganic scale applications with this technology and a generalized graph illustrating such is provided in Figure 13. Yet another laboratory study was performed for an "On / Off" Biocide Control and the results are illustrated in Figure 14. Finally the overall layout of the pilot cooling system and all the sensory equipment in the laboratory is shown in Figure 15.



Fig. 12 Delta Temp ISO vs Biofilm Thickness.



Fig. 13 Generalized Fouling Trend with Ultrasound Thickness Detection.



Fig. 14 Biofouling and Biocide-Biodispersant Addition.



Fig. 15 Pilot Cooling System and Monitors.

FIELD APPLICATION AND VALIDATION

Once the internal laboratory proof of concept work had been completed, the fouling monitors were placed into field application service. During this period their calibrations were developed and the performance cross checked with past laboratory results. A case history associated with a biofouling application validation run will be discussed herein.

Heated-Bio-Fouling Monitor

A chemical processing plant had excessive bio-fouling of the cooling tower film fill, heat exchanges and transfer lines. The make-up water was river water, which contained suspended solids and microbiological activity, plus process condensate water, which contained COD and sources of ammonia. The biological control program consisted of hydrogen peroxide slug fed every 7 days and stabilized bromine fed continuously. The monitor was placed on the cooling system return water to the cooling tower to obtain a performance baseline. Figure 16 and 17 reveal the fill fouling and the monitor's output screen respectively. It can be seen that the biofilm thickness measured was 1.211 mm (i.e., 1211 μ m). The screen thicknesses n1, n2, and n3 in Figure 17 have been renamed; scale, organic and biofilm respectively.



Fig. 16 Cooling Tower Film Fill Fouling.

2015-07-31	SOLG	2015	12:38:40
7-1	Thickness_n1	0.001 mm	8
and the second s	Thickness_n2	0.000 mm	
	Thickness_n3	1.211 mm	
	Thickness_p1	1.535 mm	
	Bulk_Temp	20.75 C	
. D	elta_Temp_ISO	5.30 C	\otimes
•	Flow	15.11/m	in 🦱
	Power	2.08	9
Soft. Serl	I. TEST	-	MENU

Fig. 17 Heated Bio-Fouling Monitor Screen.

A new biological control program was implemented that consisted of a non-oxidizing biocide, Dibromonitrilopropionamide (DBNPA, Organic Treatment 1) to be slug fed every 10 days at 17.5 ppm active, while chlorine dioxide was fed continuously to 0.3 ppm FAC (Free Available Chlorine). The chlorine dioxide would be raised to 1 to 1.2 ppm FAC twice per week for 4 hours. The results were only slightly better than prior, in that, it took longer to reach biofilm thicknesses of 2 mm. The results from the monitor are graphically displayed in Figure 18. While the 10 day biofilm is seen on the ultrasound transducer (Figure 19) and the heated stainless steel target (Figure 20).





Fig. 19 Ultrasound Probe Fouled with Biofilm.





Performance improvements were desired, so a biocide efficacy study was done the cooling water and it was found that a quaternary ammonium chloride compound (Organic Treatment 2) out- performed the DBNPA, so it was utilized at a dosage of 19 ppm active. The monitor's graphical results (see Figure 21) reveal the biofilm thickness has been held down below 0.15 mm (150 μ m). But if you look closely, it can also be seen that this performance is not consistent as the frequency of addition is shrinking from 15 days to 8 days to 6 days. The next step in performance improvement is to increase the concertation of the twice weekly ramped –up (i.e., super shot) chlorine dioxide feed and a decrease in contact time.

Fig. 18 Organic 1; Foulant Temperature Difference and Thickness.



Fig. 21 Organic 2; Foulant Temperature Difference and Thickness.

SUMMARY

Extending the capabilities of deposit monitor from exsitu crystallization detection (i.e., OnGuard[™] 3S analyzer) to insitu crystallization detection (i.e., OnGuard[™] 3H analyzer) involved more research, engineering and design challenges. Laboratory results associated with the final design revealed, that under equivalent shear stress, the calculated fouling factors associated with the inorganic scale formed were the same as those calculated with the annular heat transfer test section. Calculated fouling factors of both, the annular and new monitor had good correlation to the ultrasound calculated thickness, and those thicknesses where equivalent to physical micrometer measurements. A detection level down to below 5 µm of thickness has been confirmed. The detection process also demonstrated it can be used to activate chemical treatment to control inorganic scale deposition and to remove the same.

Additional research resulted in the development of a method to determine between hard crystalline scale and soft biofilms. The use of various organic materials as surrogate biofilms resulted in further deposition differentiation. A process was developed to apply ultrasonic amplification (i.e., higher signal to noise ratio) which enabled weaker reflected signals to be detected. Biofilm thickness calculations by ultrasound were found to be in good correlation with optical coherence tomography (OCT) down to 25 µm and at or below 10 µm, the signal was too weak for good correlations. However, the initial growth phase is still captured by the heat transfer reduction measurement. The newer advancements in the OnGuard[™] 3H analyzer, resulted in this design, now designated as OnGuard[™] 3B analyzer, and has allowed for differentiation of the type of deposit present on the heated surface. This newer process has been applied in the field to detect biofouling and optimize the biocide control program very effectively.

Not only is this new design smaller than a conventional annular heat transfer test section unit, but it requires less water flow and heat input to operate. It also provides equivalent detection results to the annular design with less heat transfer surface cross section area. That is 396 mm² compared to 4645 mm², which will result in quicker times to detect fouling occurrence. While both are far faster than a well instrumented heat exchanger, that could be up to or exceeding $13.9 \times 10^9 \text{ mm}^2$ surface area such as a utility condenser.

CONCLUSIONS

The need to progress from self-scaling detection to simulation of heat exchanger fouling and detection of inorganic scale, organics and biological matter has been successful. It has also opened up other opportunities for deposit differentiation / determination of categorical type. This newer design monitor is able to simulate surface temperature and shear stress of heat exchangers which are to be studied / evaluated for heat transfer performance improvements. It will provide faster fouling detection and can measure down to 5 µm of deposit thickness. Fast detection means the deposition has not aged sufficiently to dehydrate, as in scales, or cross-link sufficiently, as in biofilms, and accumulate other system fluid debris, all of which increase the difficulty of an on-line removal from either chemistry or chemical concentration changes and to subsequently maintain the surfaces in an adequately clean state. By such avoidance of these conditions, the foulant is not irreversible and on-line cleaning can be done rather than off-line cleaning with harsh chemicals and or physical cleaning methods. Over-all results are equivalent to the conventional annular heat transfer test section found in the OnGuard[™] 2-plus analyzer, which is NACE International RPO 189 compliant and allows for it to support validation of hydrothermal stress coefficient calculations in HexEval evaluations (Beardwood, 2015).

The newer monitors can also be used in performance and knowledge based control systems for cooling water. Utilizing the detection (KPI; key performance indicator) and distinction of the type of foulant present, proper selection of changes of the cooling water key operating indicators (KOI's), such dispersant type, concentration, as well as system cycles of concentration, pH and ORP can be adjusted if needed for the particular contingency plan implementation, to return the KPI to the set point. The robust, but compact design makes the monitor suitable for transporting and installations to and from cooling water facilities as well as within said facilities.

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NOMENCLATURE

Fouling Factor	-	(Heat Transfer Coefficient) ⁻¹
	-	hr ft ² °F / Btu X 5.674 = m^2 K / W

Watts	-	W = 3.4129 Btu / hr
Area	-	square feet; $ft^2 = 9.29 \text{ X } 10^{-2}$
square meter (m ²)		
Velocity	-	feet per second; ft / sec
	-	meters per second; $m/sec = 3.25$ ft
		/ sec
Temperature	-	Degree Fahrenheit; °F
	-	Degree Celsius; $^{\circ}C = (^{\circ}F - 23) X$
		5/9
	-	Degree Kelvin; 1° K = -475.886°F
		= -272.15°C
Weight	- kil	ograms (kg) = 2.2 pounds (lbs)
Thickness	- mie	crometer (μ m) = 0.001 mm =
0.000001 m = 1 mic	ron (μ) = 0.0004 inches

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