Vol. 6

No. 1

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# LABORATORY METHODS FOR THE EVALUATION OF TREATED WASTEWATER AND THEIR INDUSTRIAL USABILITY

The paper presents laboratory methods for the estimation of industrial usability of the treated wastewater. These methods are based on principles of modelling and enlargment of the scale of industrial processes. They allow to determine the number of the admissible condensations in open recirculating systems, the sedimentation rates of soft and hard deposits and the corrosion rate. The necessary equipment and the way of its application to recirculating open systems and once-through systems have been described. The equipment and methods of estimation of the treated wastewater usability for other than cooling purposes have been also described.

#### 1. INTRODUCTION

In view of the deficiency of natural water resources [3] the reclamation of water and industrial utilization of treated wastewater belong to the most important problems connected with water supply. A qualitative evaluation of the treated wastewater is performed by means of suitable tests. Of the total consumption of water used by industry the highest percent (60-70) falls to the cooling of technological equipment [9]. Smaller amounts fall to hydrotransport, washing, rinsing etc. The basic characteristics required from industrial water, the cooling one in particular, are the following:

the lack of sedimentation and scale-formation properties; the lack of corrosive interaction with ambient materials.

In the case of water used for cooling purposes, surface cooling in particular, the above requirements should be satisfied in the presence of high heat loads of the surface cooled.

The paper presents two methods by which the industrial usability of the treated wastewater can be evaluated. The first method is applied when the treated wastewater is used for the surface cooling of industrial aggregates, while the second one finds its application in the case of non-heated water.

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<sup>4 -</sup> Environment Protection Engineering VI/1

# 2. BASIC ASSUMPTIONS CONCERNING THE METHODS OF WATER TESTING FOR THE SURFACE COOLING OF INDUSTRIAL AGGREGATES

According to the most recent nomenclature the following systems of surface cooling [1] are distinguished:

once-through in which the cooling water flows only once through the unit being cooled; open recirculating systems in which the water circulates many times through the aggregate being cooled, but the water jet is stopped in the cooler where the water, being brought into contact with air, partly evaporated and is condensed;

closed recirculating system in which the water circulates within a closed system; it has no direct contact with air and does not evaporate.

The treated wastewater may be used mainly in the first two kinds of the systems described.

One of the basic parameters characterizing the surface cooling process is heat load q of the surface being cooled [6]. Heat flux which penetrates the cooling water while passing through the cooled wall meets a number of resistances that decide upon the drops of temperatures occurring along the distance travelled by the heat flow. In this case, the essential parameter of the cooling process is the temperature of the interfacial water layer on the wall of the element being cooled. This temperature may be assumed as being approximately equal to that of the wall of the element cooled. It is among others related functionally to the water flow rate. This problem is treated in detail in the theory of thermal motion [2]. When the heat load is known or given, the wall temperature of the element cooled and of the interfacial water layer can be calculated from the formula

$$t_{w} = \frac{\overset{*}{q}}{a_{2}} + t_{m},$$

$$t_{w \max} = \frac{\overset{*}{q}}{a_{2}} + t_{\max},$$

where:

 $a_2$  — heat penetration coefficient which is the function of Reynolds, Nusselt and Prandl numbers; it is calculated according to the procedure given in the theory of heat motion [2]:

 $t_m$  or  $t_{max}$  — the mean or maximal temperatures of circulating water.

In the case of open recirculating systems the circulating water is partly evaporated in the cooler and is condensed. The condensation degree can be calculated from the balance of losses occurring in the system [7]

$$n_{gr} = \frac{w_p + w_r + w_a}{w_a + w_r} = w_p \left(\frac{1}{w_r + w_a}\right) + 1,$$

where:

 $w_n$  – evaporation losses in the system,

 $w_r$  - spatter losses in the cooler,

 $w_c$  — losses due to the system desludging.

The amount of make-up water required to compensate the circulating water is equal to the sum of the losses:

$$w_p = w_p + w_r + w_a.$$

If the cooling system is not desludged then,  $w_a = 0$ , and the condensation number takes its maximum value:

$$n_{\max} = w_p \left( \frac{1}{w_r} \right) + 1.$$

To avoid the excessive condensation of circulating water which would enhance the sedimentation, scale formation or increase the corrosion rate, the condensation degree of circulating water cannot exceed the value of the admissible condensation number  $n_d$ . It can be determined according to the method described below. Knowing the value of  $n_d$  it is easy to calculate the values of indispensable system desludging  $w_a$  and the consumption of make-up water  $w_d$ :

$$w_a = \frac{w_d}{n} - w_r,$$

$$w_d = \frac{n_d}{n_d - 1} w_p.$$

In order that the investigations performed in laboratory scale give information valid on an industrial scale, the model conditions resulting from the change of the experimental scale should be kept invariant [4]. This, in particular, refers to: heat loads of the full scale unit and laboratory unit, wall temperatures of the elements cooled in the full scale unit and laboratory unit, water flow rate which in industrial plant should be the same as in the laboratory model.

If these conditions are satisfied then the average and maximum water temperatures as well as its flow rate in laboratory model can be calculated [8].

The investigations performed according to the above program allow to determine experimentally a number of indices on the basis of which the industrial usability of the treated wastewater can be evaluated. These are the following:

sedimentation rate of the soft deposits in  $g/m^2h$  or mm/y, sedimentation rate of hard deposits (scale) in  $g/m^2h$  or mm/y,

corrosion rate of the selected materials (steel, brass etc.) in g/m²h or mm/y.

If the water is to be used for the compensation of losses occurring in opened recirculating systems, the experiments give the additional information, namely: the value of admissible water condensations and chemical composition of circulating water.

# 3. EXPERIMENTAL EQUIPMENT FOR THE EVALUATION OF WATER USABILITY IN OPEN RECIRCULATING SYSTEMS

The design of experimental equipment for model testings was based on the principles of a reduced-scale modelling of technological processes [4], [8]. (It was patented as a functional pattern under number 22288 and presented in figs. 1 and 2.) Circulating water, pumped by pump (3), flows through the measuring devices: glass vessels with test plates for tests of sedimentation and corrosion rates (10) (fig. 3) and a heater (6) where it is heated

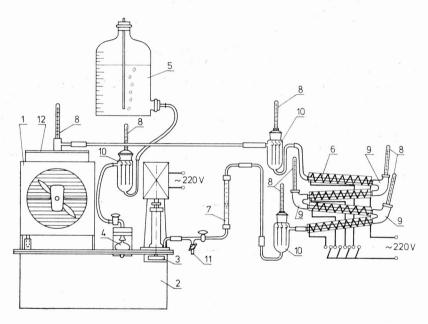


Fig. 1. Scheme of a model laboratory set-up

1 - cooler, 2 - tank of cooled circulating water, 3 - pump for circulating water, 4 - float chamber, 5 - bottles with make-up water, 6 - heater, 7 - rotameter, 8 - thermometers, 9 - elbow connections, 10 - vessel with test plates, 11 - water tap, 12 - hot water tank

#### Rys. 1. Schemat laboratoryjnego zestawu modelowego

1 - chłodnia, 2 - zbiornik wody obiegowej chłodzonej, 3 - pompa wody obiegowej, 4 - komora pływakowa, 5 - butle z wodą dodatkową, 6 - nagrzewnica, 7 - rotametr, 8 - termometry, 9 - kolanka łącznikowe, 10 - naczyńka z płytkami testowymi,
 11 - kurek do poboru próbek wody, 12 - zbiornik wody gorącej

to the desired temperature. Hot water flows to the tank (12) whence it trickles on the cooler's sprinkler (1) (fig. 4), and being cooled by evaporation it flows to the tank (2). Make-up water flows from the bottle (5) through a float regulator (4) (fig. 5) which maintains a constant water level in the float chamber and a constant volume of circulating water in the equipment.

The heater (figs. 6, 7) is a particularly important element of the system. Its kind and size are selected according to model conditions corresponding to the standard, i. e. to the

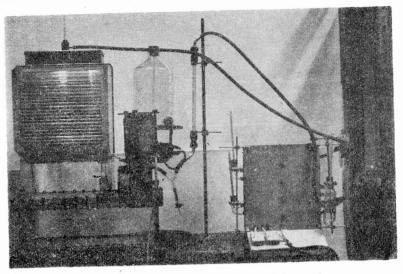


Fig. 2. Model laboratory set-up ready to work Rys. 2. Laboratoryjny zestaw modelowy przygotowany do pracy

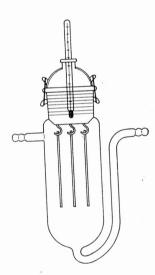


Fig. 3. Flow vessel with test plates

Rys. 3. Naczyńko przepływowe z zawieszonymi

płytkami testowymi

industrial plant. The most convenient are electric heaters which allow the control of heat load and — what is particularly important — the application of short, easily weighed metal pipes. The acid-resistant steel pipes allow to determine the sedimentation rate in examined water. Application the pipes of steel and chromium plated outside allows to determine steel corrosion rate in water examined under heat load and dynamic conditions. If necessary, the pipes can be made of brass, copper etc

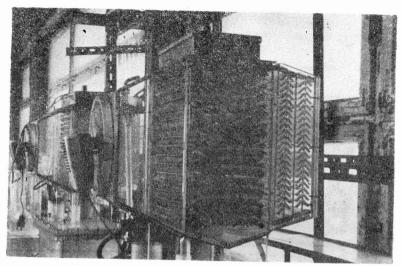


Fig. 4. Side view of the cooler showing the construction of spray elements and the bleeder-type spray condensor

Rys. 4. Widok chłodni z boku w celu uwidocznienia konstrukcji elementów ociekowych i odkraplacza trójrzędowego

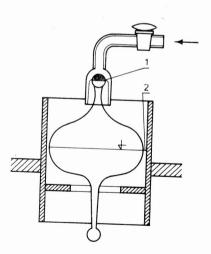


Fig. 5. Chamber with float regulating the make up water inflow Rys. 5. Komora z pływakiem regulującym dopływ wody dodatkowej

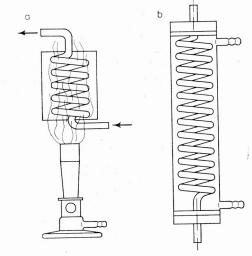


Fig. 6. Heaters

spiral heated with a gas burner, b - spiral heated with a steam at 100 °C or hot oil

#### Rys. 6. Nagrzewnice

- spiralna ogrzewana palnikiem gazowym, b - spiralna ogrzewana parą wodną o temperaturze 100 °C lub gorącym olejem

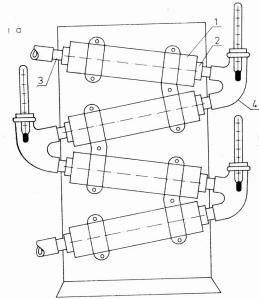


Fig. 7. Electric heater

1 - heat insulation with resistance wire, 2 - ceramic pipe, 3 - steel pipe, 4 - elbow connections with a thermometer

### Rys. 7. Nagrzewnica elektryczna

- 1 otulina z drutem oporowym, 2 rurka ceramiczna, 3 - rurka stalowa, 4 - kolanka łącznikowe z termo-
- metrem

## 4. EXPERIMENTAL METHODS AND DISCUSSION OF RESULTS

In order to get a full physicochemical characteristic of the water examined, two cycles of tests should be performed.

In the first cycle, the experiment is conducted without the desalting of circulating water in order to obtain its possibly high (n = 6-10) condensation. This cycle of experiments lasting for 190–200 h was intended to determine the number of admissible condensation  $n_d$ .

In the second cycle, the water condensation is proceeded up to the value of admissible concentration determined earlier, and while the circulating system is desludged this condensation is kept invariant during the whole experiment (200-300 h). The purpose of this cycle is to determine the sedimentation rates of soft and hard deposits and the corrosion rate of the materials examined. In each cycle, the samples of water for analysis are taken within equal time intervals to determine the concentrations of chlorides and sulphates, the pH values, alkalinity in the presence of methylorange "m", phenolphtalein "p" as well as total hardness etc.

Experimental cycles should be conducted in continuous way (three shifts of servicing personnel). Thereupon a total analysis of circulating water, including its thermostability, should be perfored [5].

Metal pipes of the heater, being taken out from ceramic casings, are dried and weighed, then the accumulated deposits are removed by a careful etching with a suitable solution (eg. 10% HCl with tardial and thiourea). The incrustation  $\Delta g_1$  and material loss due to corrosion  $\Delta g_2$  are calculated from the differences in the weights of pipes:

$$\Delta g_1 = g_1 - g_0,$$
  
 $\Delta g_2 = g_0 - g_2,$ 

where:

 $g_0$  — initial weight of the pipe (g),

 $g_1$  — weight of the pipe at the end of the cycle (g),

 $g_2$  — weight of the pipe after the deposit is removed by etching (g).

A similar procedure is applied to test plates. They are dried and weighed after being taken out from the apparatus. Deposits easily removable are washed down under a weak stream of water, using a soft brush, and the pipes are dried again. Thereupon the plates are subject to etching to remove hard deposits. This procedure allows to determine:

the total increment of deposits

the amount of soft deposits 
$$\Delta g_1' = g_1' - g_0',$$
 the amount of hard deposits 
$$\Delta g_2' = g_1' - g_2',$$
 the amount of hard deposits 
$$\Delta g_3' = g_2' - g_3',$$
 loss due to corrosion 
$$\Delta g_4' = g_0' - g_3,$$

where:

 $g_0'$  - initial weight of the plate,

 $g_1$  — weight of the plate at the end of the experimental cycle,

g'<sub>2</sub> - weight of the plate when soft deposits are washed down,

 $g_3'$  — weight of the plate after etching.

These values, referred to the duration of the experiment, represent the rates of the parate processes. Results from the analysis of the circulating water samples, taken during the first experimental cycle, are transformed into momentary numbers of condensation:

$$n_i = \frac{C_i \text{ circul.}}{C_{i_0}},$$

here

 $C_{i_0}$  — initial concentration of the component i in circulating water,  $C_{i \, {\rm circul.}}$  — concentration of the component i in circulating water after a time  $\tau$ .

The calculated numbers of condensation are plotted in coordinates  $n_i - \tau$  (fig. 8). The action  $n_i = f(\tau)$  for a component readily soluble in water, e.g.  $n_{\rm Cl}$ — is represented by straight time with a constant inclination angle. For other component, e.g.  $n_m$ ,  $n_{tw}$  etc.,

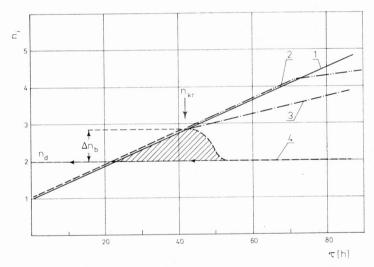


Fig. 8. Exemplary plot of treated wastewater condensation  $n_{\text{Cl}^-} = f(\tau)$ , 2 - function  $n_{m} = f(\tau)$ , 3 - function  $n_{\text{SO}_4}^{-2} = f(\tau)$ , 4 - function  $n_{tw} = f(\tau)$ 

Rys. 8. Przykładowy wykres przebiegu procesu zagęszczenia wody odzyskanej ze ścieków 1 – funkcja  $n_{\text{Cl}^-} = f(\tau)$ , 2 – funkcja  $n_{m} = f(\tau)$ , 3 – funkcja  $n_{\text{SO}_4^{-2}} = f(\tau)$ , 4 – funkcja  $n_{tw} = f(\tau)$ 

which can precipitate from the solution, the corresponding curves follow initially the ourse of that for  $n_{Cl}$ , but later on they are broken, giving evidence to various reactions ccurring in circulating water. For example, the curve  $n_m$  (fig. 8) after reaching the value is broken and eventually runs parallelly to the X-axis. The course of curve indicates that the condensation of water increases until the critical value is achieved. At this moment recipitation of carbohydrates starts. The horizontal course of the curve corresponds to the equilibrium between the carbonates fed in with water and those precipitated from the solution. Shaded area represents the transitory oversaturation. It, however, does not

appear always, being dependent on the water buffer capacity. By extrapolating the horizontal branch of the curve  $n_m$  to the left we find the sought number of admissible condensation  $n_m$ .

The difference

$$n_b = n_{kr} - n_d$$

is the numerical value of the water buffer capacity at the supersaturation state.

# 5. EXPERIMENTAL EQUIPMENT AND THE METHOD FOR INVESTIGATION O ONCE-THROUGH CIRCULATION

To estimate the usability of treated wastewater in the case of once-through system fo cooling, the heater alone is used to which the water is delivered by a suitable pump. The experiment is conducted in the continuous manner for 100–200 h. It allows to determine the sedimentation and corrosion rates in the presence of heat load of the surfaction cooled (heater's pipes) and without it, but in the latter case at two different temperatures (on test plates).

# 6. EXPERIMENTAL EQUIPMENT AND THE METHOD OF QUALITATIVE ESTIMATION OF THE TREATED WASTEWATER WITHOUT HEAT LOAI

In this case the simple equipment is used, which can be enlarged arbitrarily to include a greater number of stands (fig. 9). A single stand is shown in fig. 10. The equipment allow to determine the sedimentation rates of soft and hard deposits and the rate of corrosio

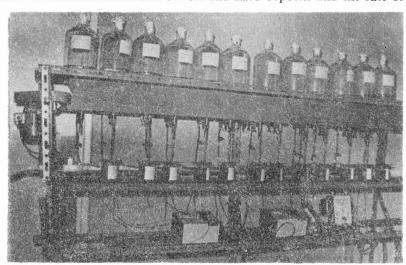


Fig. 9. Multiple-stands set-up for determining of corrosion and sedimentation rates

Rys. 9. Wielostanowiskowy zestaw do wyznaczania szybkości korozji i odkładania sie osadów

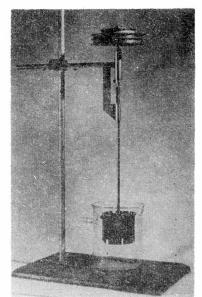


 Fig. 10. Single set-up for determining of corrosion and sedimentation rates
 Rys. 10. Pojedynczy zestaw do wyznaczania szybkości korozji i odkładania się osadów

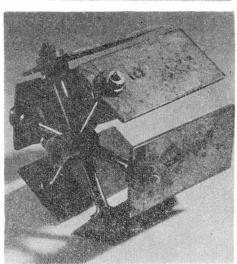


Fig. 11. Holder fastening test plates Rys. 11. Uchwyt do umocowania płytek testowych

at room temperature, as well as at elevated temperatures when a thermostate is applied. Six test plates are hanged on an appropriate acid-resistant steel holder (fig. 11) and insulated electrically from the holder by foil pads. The whole equipment is driven by an electric motor, and the rotational speed of plates satisfies the basic criterion of similarity

$$U_m = U_w$$

i.e. peripheral speed  $U_m$  of the plate must be equal to the flow rate  $U_w$  of the water in the industrial unit.

The water examined is continuously fed to beakers in which the plates are immersed and later it is discharged through an overflow to a sewer. In this way the water is continuously renewed. The construction of the apparatus allows to apply additional treatments such as water aeration, deoxidation by nitrogen blowing etc. The experimental cycle lasts c. 6 weeks. The separate test plates are taken out consecutively in one week intervals, then they are weighed, dried, sediments are removed etc. The whole procedure is the same as that previously described. It is convenient to replace the plates with the new ones as then the results are doubled. The values of sedimentation and corrosion rates determined in this way are plotted using  $v_i$  and  $\tau$  as coordinates. From the curves we determine the initial and established rates of separate processes as well as the time after which the processes proceed at a constant rate (fig. 12).

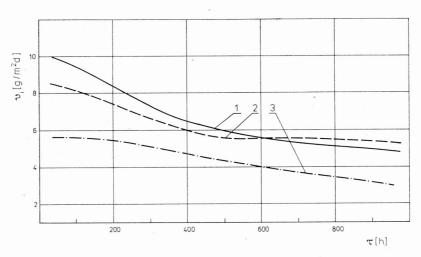


Fig. 12. Exemplary plot of the sedimentation and corrosion rates

1 – corrosion rate, 2 – soft deposit sedimentation rate, 3 – hard deposit sedimentation rate

Rys. 12. Przykładowy wykres przebiegu krzywych szybkości odkładania się osadu i korozji 1 – szybkość korozji, 2 – szybkość odkładania się osadów twardych

#### 7. CONCLUSIONS

The methods presented have been applied several times in order to estimate both the industrial usability of the treated wastewater and surface water as well as various methods of their treatment or correction. It should be noticed that the methods described allow to obtain the necessary data under operating conditions of the given industrial establishments. It is of particular importance in design and modernization of industrial plants.

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### LABORATORYJNE METODY OCENY PRZYDATNOŚCI WÓD ODZYSKIWANYCH ZE ŚCIEKÓW DLA CELÓW PRZEMYSŁOWYCH

W artykule przedstawiono laboratoryjne metody badania przydatności dla celów przemysłowych wody odzyskanej ze ścieków. Podstawą tych metod są zasady modelowania i powiększania skali procesów przemysłowych. Metody te pozwalają na wyznaczenie liczby zagęszczenia dopuszczalnego w otwartych cyrkulacyjnych obiegach chłodzących, szybkości odkładania się osadów miękkich i twardych oraz szybkości korozji.

Opisano niezbędną aparaturę i sposób jej stosowania dla obiegów chłodzących cyrkulacyjnych otwartych i obiegów chłodzących przepływowych. Ponadto opisano aparaturę i omówiono metody badania przydatności wody odzyskanej ze ścieków stosowanej dla celów niechłodniczych.

### LABORMÄSSIGE METHODEN ZUR BEWERTUNG DER WASSERRÜCKGEWINNUNG FÜR INDUSTRIELLE ZWECKE

Der Beitrag erläutert Labormethoden, die für die Bewertung der Wasserrückgewinnung dienen. Die Methoden stützen sich auf den Grundlagen der Modellierung und Maßstabvergrösserung der Industrieverfahren. Sie gestatten die Berechnung der zulässigen Eindickzahl in offenen Kühlverfahren, der Ablagerungsgeschwindigkeit von kristallinischen und amorphen Schlämmen sowie der Korrosionsgeschwindigkeit. Beschrieben wird die Apparatur und ihre Abänderungen für verschiedene Kühlwasserkreisläufe. Beschrieben und erklärt wird auch die Apparatur zur Bewertung des rückgewonnenen Wassers für andere industrielle Zwecke.

### ЛАБОРАТОРНЫЕ МЕТОДЫ ОЦЕНКИ ПРИГОДНОСТИ ВОД, РЕГЕНЕРИРОВАННЫХ ИЗ СТОКОВ, ДЛЯ ПРОМЫШЛЕННЫХ ЦЕЛЕЙ

В статье описаны лабораторные методы исследования пригодности воды для промышленных целей, регенерированной из сточных вод. Основой этих методов являются принципы меделирования и увеличения масштаба промышленных процессов. Эти методы позволяют определить число лопустимого содержания твёрдого в открытых циркуляционных холодильных циклах, скорость отложения мягких и твёрдых осадков, а также скорость коррозии.

Описана необходимая аппаратура и способ её применения для открытых циркуляционных холодильных циклов, а также проточных холодильных циклов. Кроме того, описана аппаратура и обсуждены методы исследования пригодности воды, регенерированной из сточных вод, применяемой для других, нехолодильных целей.