CLEANING OF SMALL COMPONENTS OF COMPLEX GEOMETRY BY MEANS OF THE SODIUM-ALCOHOL REACTION

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Abstract

The results of some experiments on the vacuum reaction between butylcellosolve and sodium, contained in small diameter capillaries, are reported.

The effects on the cleaning rate of the temperature, amount of solvent, diameter and position of the capillaries are analyzed.

The facility, used for the cleaning of small components of complex geometry, is described.

Introduction

The choice of a sodium removal process from plant components depends on the size and on the geometry of the component. Rather difficult is the sodium removal from components where bellows, screw threads, cavities and narrow gaps are present. In fact in these cases the solvent penetration is very difficult. In several papers (1) (2) (3) (4), experiments have been carried out to try to extend the use of water or alcohol in the cleaning of components of very complex geometric form. The rates of sodium-solvent reaction, with the sodium contained in crevices of different sizes, have been measured.

In this paper the vacuum reaction between the sodium, contained in glass capillaries and in some screw threads, and two organic solvents is studied. The organic solvents are the mixture butylcellosolve (2 butoxiethanol) - NN dimethylformamide and the solvent commercially known as CHIMEC-NR.

The dipendence of the cleaning rate on the amount of solvent, temperature, internal diameter, position of the capillary are examined.

On the basis of the results obtained from these laboratory experiments, a facility has been realised with the aim to perform a vacuum cleaning operation of complex geometry components.

Experimental part

Glass capillaries containing sodium, from 5 to 10 cm long, with an internal diameter ranging from 1 to 3 mm, have been dipped into the solvent inside a cylindrical glass apparatus thermostatically controlled, having an internal diameter of 6 cm and a height of 15 cm.

The capillaries have been positioned in the solvent vertically and horizontally.

The apparatus itself has been equipped with two traps, the first at 5°C, the second at-180°C, in order to condense the solvent vapours. The vacuum has been produced in the apparatus by a rotary pump.

The pressure has been measured between the second trap and the pump; its value was about 10^{-1} Torr.

The solvents used were the mixture butylcellosolve 90 vol%-NN dimethylformamide 10 vol%, obtained by mixing C. Erba RPE products, and CHIMEC-NR, supplied by the CHIMEC-ROMA, essentially containing butylcellosolve.

Laboratory experiments for cleaning screw threads have been carried out in the same apparatus previously described, at a temperature of 40°C, in vacuum.

Steel rods AISI 316, having a diameter from 4 to 5 mm, and screwed with steel nuts, from 10 to 15 mm high have been dipped into sodium at 400°C, in vacuum, to be sure that the metal could penetrate into the screw threads.

After 4 hours, the rods, with the fixed nuts have been removed from the sodium, cooled and dipped into the solvent.

As soon as the hydrogen bubbling stopped, the washing solvent has been discharged, and new butylcellosolve has been introduced.

The cleaning process lasted for a few minutes more, in vacuum, until the gas bubbling stopped again.

This rinsing process has been repeated three times.

Then the rods and the nuts have been separated, washed by acetone, and finally dried.

Results and discussion

The reaction rate between sodium and washing solvent inside a component depends on the capability of the solvent to come into contact with the sodium. If the sodium is contained in screw threads, bellows, narrow gaps, pipes of very small diameters, the reaction is generally very slow.

At atmospheric pressure the cleaning rate initially very high, decreases with time untill it reaches gradually very low values. For example, with capillaries of 1,5 mm as internal diameter, after 48 hours only 2 mm are cleaned, when the capillary is in horizontal position.

For a vertically positioned capillary, 12 mm of the upper side and 1 mm of the lower side are cleaned after the same time.

As the sodium is moving away from the edge of the capillary, the hydrogen produced is very hardly expelled, so that the gas bubble separates the sodium from the solvents, and the reaction stops.

It is then necessary to remove the hydrogen to avoid stopping the reaction.

If the reaction is carried out in vacuum, the gas is expelled from the capillary as soon as it is formed, and the solvent in contact with the sodium can continuously be renewed.

Figure 1 shows the results of an experiment carried out in order to compare the evolution of the reaction at atmospheric pressure and in vacuum, by plotting the millimiters of cleaned capillary vs the time.

The results refer to the upper side of the capillary, with an internal diameter of 1.5 mm, and which was in a vertical position. The solvent was the mixture butylcellosolve-NN dimethylformamide.

When the reaction is started at 25°C and at atmospheric pressure, the sodium quickly dissolves itself (segment AB in fig. 1)

During 5 hours, about 10 mm of the capillary have been cleaned, the mean cleaning rate value was about 2 mm/h.

When the vacuum is produced in the apparatus, the cleaning rate increases only until 3 mm/h (BC in fig. 1). This is due to the fact that the sodium-solvent interface is still next to the edge of the capillary and the hydrogen has no difficulty to bubble out with or without vacuum. In the stage CD we have again atmospheric pressure, with the sodium 15 mm under the edge of the capillary. Under this condition the reaction is more difficult: the cleaning rate decreases to 0.6 mm/h. Producing again vacuum condition (DE in fig. 1), the cleaning rate returns to the initial values.

However these conditions (vacuum and 25°C) are not always sufficient When the apparatus is reported to atmpspheric pressure, the reaction stops. The sodium surface is about 6 cm from the edge of the capillary. This is shown in fig. 1 (segments EF, GH, MN). It has to be noted that the reaction does not start when the vacuum is restored (FG, HI, LM). In fact the sodium dissolution is hindered by a thin layer of reaction products which are formed on the surface of the sodium. The reaction is so slow, at atmospheric pressure and at room temperature, that even after 112 hours (IL), only unmeasurable variations can be observed.

When we increase the temperature at 40°C, (under vacuum) the reaction starts again (NO, RS), with a rate of 1.6 mm/h. (The sodium surface is at about 7 cm from the edge of the capillary). It has to be noted that the vacuum is needed because at the same temperature, but at atmospheric pressure, the reaction is still very slow (OP, PQ, QR).

In conclusion, the temperature of 40°C and the presence of vacuum are the conditions to obtain the complete cleaning of the capillary. As the rate of the sodium solvent reaction always depends on the geometry of the components, we have considered useless any accurate evaluation of the reaction rate.

We have preferred, instead, to study qualitatively the dependence of the cleaning rate on the amount of solvent, the internal diameter, the position of the capillary and the temperature level.

A) Influence of the amount of solvent used

A capillary (internal diameter 1.5 mm) has been dipped vertically into the mixture butylcellosolve - NN dimethylformamide, at a temperature of 40°C in vacuum. The solvent level was about 1cm over the upper edge of the capillary.

Under vacuum, the initial cleaning rate was about 3 mm/h, until about 20 mm of the capillary were cleaned. (These data always refer to the upper side). When solvent was added, the cleaning rate increases to about 5-6 mm/h.

The change of the sodium concentration in the solvent cannot, by itself, justify such an increase of reaction rate, because the amount of sodium previously present in the solvent was very low.

The reason why the reaction rate increases is to be found in the fact that the level of the solvent is now 3-4 cm over the edge of the capillary and the penetration in the capillary itself is favoured.

The solvent comes in easier, while the hydrogen is sucked out.

B) Influence of the capillary diameter

Fig. 2 shows the cleaning rate in two capillaries of different internal diameters (1.5 and 3 mm) and of the same lenght (10 cm). In order to observe the cleaning action in the same conditions, the capillaries have been dipped into the solvent vertically at the same time. It can be observed that the 1.5 mm (I.D.) capillary is cleaned more quickly. The washing process is only regulated by the dissolution rate of the sodium, which, in the narrower tube, is contained in a smaller amount.

As the free surface of the sodium decreases within the capillary, the evolution of reaction is also influenced by the size of the capillaries themselves, in spite of the presence of the vacuum. Successively then, in the narrower capillary, the cleaning rate decreases, while, in the larger one, it remains almost constant.

C) Influence of the capillary position

The position of the capillaries is the factor which has the most evident influence on the cleaning rate. Fig. 3 shows the sodium dissolution rates in two capillaries (I.D. 1.5 and 3 mm) horizontally placed in the cleaning apparatus.

At the beginning and until sodium surface is about 10 mm from the edge of the capillary, the cleaning rate, is almost the same in both capillaries (1.5 mm/h) but soon after it decreases to values lower than 0.5 mm/h. Such values are significatively lower than those observed in vertically positioned capillaries. While at atmospheric pressure the the reaction stops, in vacuum, on the contrary, a slow hydrogen release is observed also after 28 hours.

When the capillaries are in vertical position the reaction, even if very slow, does not stop neither in the lowest part of them.

D) Influence of the temperature

The cleaning rate at 40°C and 60°C under vacuum has been studied only with the CHIMEC-NR as solvent. (the mixture butylcellosolve-dimethylformamide decomposes at 40°-50°C (5)).

Two capillaries (internal diameter 1.5 mm) have been dipped vertically into the same quantity of solvent. The capillary, about 10 cm long, has been washed at the temperature of 60°C in about 13 hours with a mean rate of about 8 mm/h. At the temperature of 40°C the cleaning rate is smaller (about 5 mm/h). In both cases the rates are constant, also when the sodium surface is far from the edge of the capillary.

Purification of screwthreads

The sodium removal from screw threads is one of the most difficult problems of components cleaning. In these cases an incomplete washing can be the cause of dangerous seizing phenomena. Satisfactory results, in removing the sodium retained in some screw threads, have been obtained by using the vacuum cleaning technique. After 30 hours the sodium has been completely removed from 4 screw threads, obtained from steel rods of the diameter of 4 mm.

After the cleaning the nuts have been separated from the rods: no sodium has been observed on the screw threads, and no trace of sodium carbonate appeared on them, after several days of storage in the open air. On two of the screw threads obtained from rods with 5 mm of diameter, sodium traces were still present on the central part, even after a cleaning lasting 50 hours. Consequently an hydrogen production has been still observed. Despite the incomplete cleaning of the two last screw threads, we think that the results of these experiments are rather satisfactory.

Purification of small components of complex geometry

On the basis of the above results a facility has been realised to perform the cleaning of small components of complex geometry (fig. 5). These components cantained sodium in parts hardly reachable by a simple dipping into the solvent at atmospheric pressure.

The block diagram of the facility is shown in fig. 4.

The components to be cleaned are placed in the container S_2, where the vacuum is made by means of a rotary pump P. The container consists of a steel cylinder, 50 cm high, with an internal diameter of 30 cm. The opening of the valve V allows the solvent to flow from the feed tank S_1 to the cleaning vessel S_2. The solvent vapours are condensed by means of two traps, the first at 5°C, the second at liquid air temperature. The reaction is controlled by the measurement of the developed hydrogen with a nikel membrane detector, assembled in our laboratory (6). The use of a

very sensitive detector is important, in these cases, because the sodium solvent reaction is generally very slow, and the hydrogen quantity to detect is very small. When the hydrogen production stops, the solvent used is drained and substituded by a new solvent. This rinsing operation is carried out in vacuum to allow the solvent to penetrate in all narrow gaps. The rinsing action is repeated several times, until no sodium traces are to be found in the drained solvent. Therefore the components are rinsed in vacuum with deionized water, then with acetone and finally dried. During the water rinsing stage, we never observed any hydrogen release.

The above procedure, which uses CHIMEC-NR as solvent at 40°C, has been applied to clean the following small components:

- Some pipes, whith an internal diameter 5-6 mm, 30 cm long;
- Some Gachot bellow valves;
- Specimen holder of the test section and plug meter of the experimental loop C S 5 of the C.S.N. Casaccia;
- Duct of a flow meter;
- A steel poral filter, welded within a steel container.

The components disassembled or sectioned after the cleaning process show no trace of sodium.

In the case of the valves, some difficulties have been observed. In fact the solvent, penetrated through the upper side seals of the valve, has damaged the mechanical part which act the opening and the closing of the valve itself. This difficulty has been however overcome by the use of a simple apparatus, where the valves are cleaned without the need of dipping them into the solvent. The valve, closed at one side, has been connected by a joint to a solvent container, where the vacuum can be produced.

Conclusion

The results obtained applying the vacuum cleaning process are encouraging and show that it is possible to use the butylcellosolve to remove with success the sodium from small and geometrically complex components.

Experimental work to test the vacuum cleaning processes on models reproducing the geometries of some mechanism of the PEC reactor (Fuel elements testing reactor) is under way and the results will be available in the next future.

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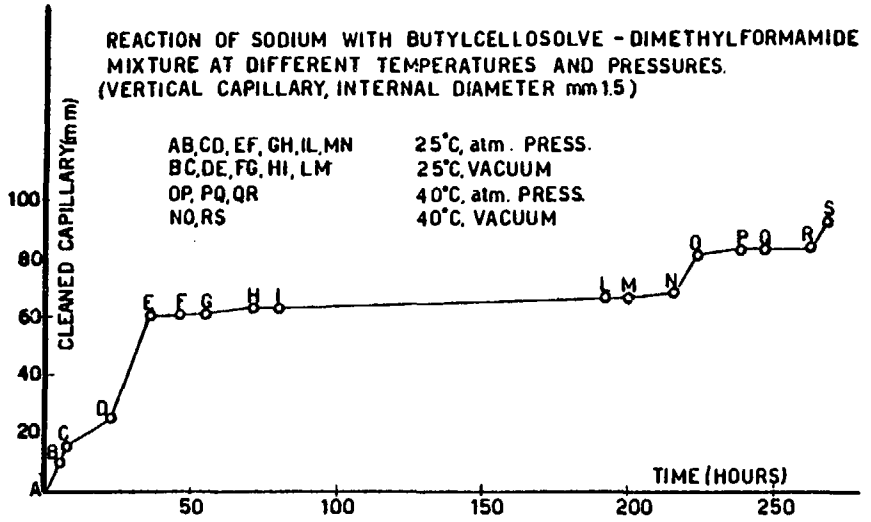

Fig.1

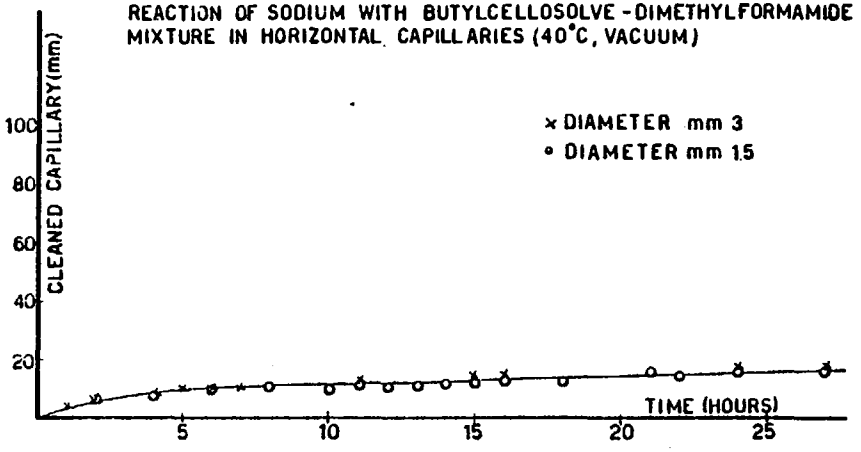

Fig.3

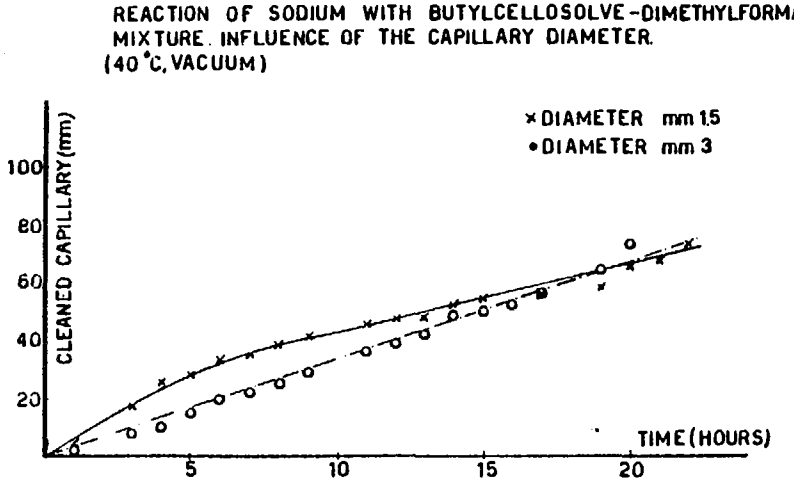

Fig.2

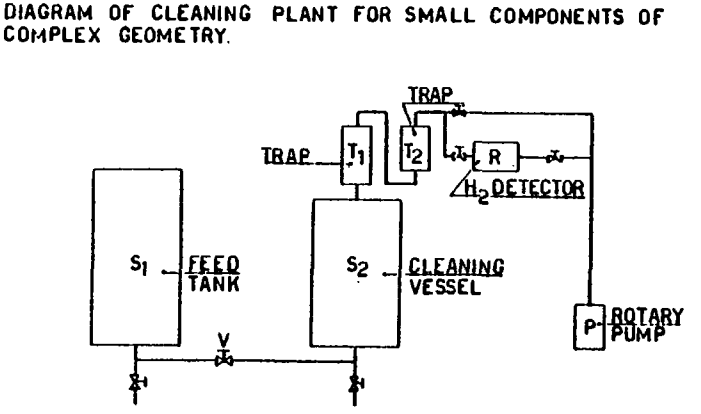

Fig.4

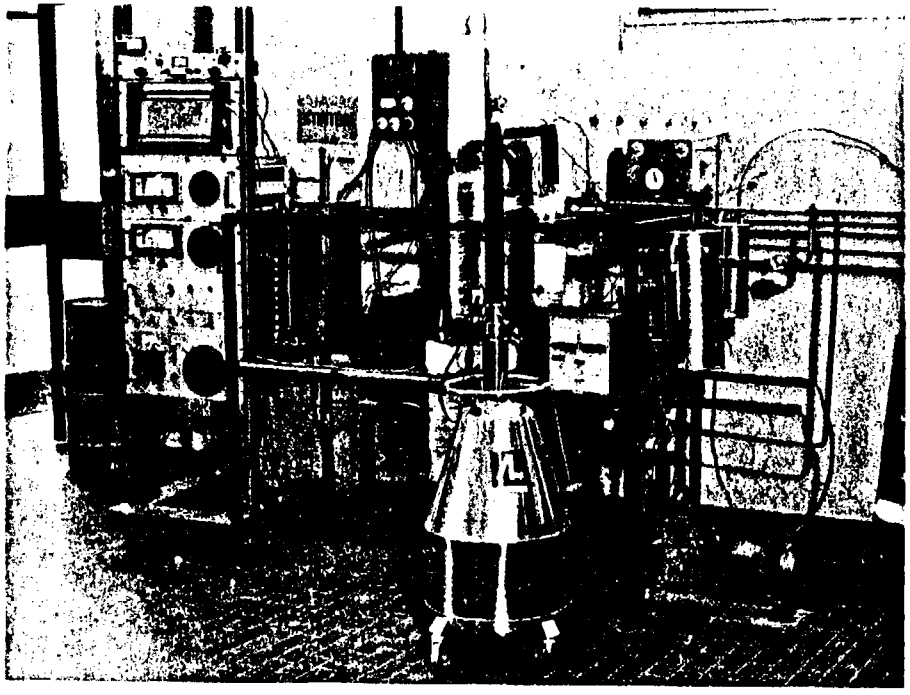

FIG. 5 SODIUM CLEANING LABORATORY FACILITY

SODIUM COMPONENTS CLEANING STATUS IN THE ITALIAN FAST REACTOR PROGRAM

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As a consequence of the Italian Fast Reactor Development, mainly aimed to the PEC project and to the partecipation in the French Superphenix project, it is of increasing importance to set up a reliable method of sodium removal for specific reactor components and related test loops.
So far the problem has been faced only in few occasion and in any case without contamination.
The first problem has been the cleaning of the PEC Fuelling Machine (MCS), that has been ordered by NIRA, PEC main contractor, to the English firm GEC REL, and whose prototype has been already built.