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SLUDGE CHARACTERIZATION AND STANDARDIZATION METHODS: DEVELOPMENT, STATUS, TRENDS

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ABSTRACT

A short history of sludge characterization is given leading from sewage and sewage sludge treatment in former times to modern water and soil pollution control with resulting needs of sludge investigation and analysis. The progress in this field in the Federal Republic of Germany from the sixties till today is given as an example and the connections with the development in Europe with special regard to the concerted Action COST 68/681 of the Commision of the European Communities between 1972 and 1990 are described. Finally, the present situation is delineated which is determined by the work of the CEN Technical Commitee 308 "Sludge characterization", which was founded in May 1993.

KEYWORDS

Sewage sludge, sludge characterization, sampling, sludge analysis, standardization methods.

INTRODUCTION

As long as the collection of sewage and wastewater in sewerage systems and their handling in treatment plants of municipalities or water boards is performed, problems of characterizing wastewater and sludge still exist. In earlier times there were predominant needs for process control of biological wastewater treatment and sludge handling, which was mainly anaerobic digestion and dewatering. Here the settling characteristics of the activated sludge suspension, the water/dry matter content of the settled or thickened sludge and the pH value at various process stages were determined. At these times sewage sludge was more or less estimated to be a by-product of sewage treatment and disposal.

DEVELOPMENT OF SLUDGE CHARACTERIZATION

At the end of the fifties and during the sixties sewage treatment in central Europe gained greater importance. Caused by problems of incomplete bio-degradation of surfactants from detergents, which had led to intensive foaming on surface waters during the dry summer of 1959, an improvement of wastewater treatment started, leading to the establishment of biological wastewater treatment facilities at nearly all important treatment plants. With the improvement of sewage treatment, the amount of sewage sludge increased remarkably and became an important technical and financial factor in wastewater technology. A large amount of sewage sludge went to agriculture and, where agricultural use was difficult or impossible, an uncontrolled disposal on land or into the sea often took place.

Despite different efforts and research work done in well-known technical and scientific institutions, a scientifically orientated sludge characterization was – in contrast to water and wastewater analysis – at this time more or less a business of specialists. Some very simple methods of sludge investigation – as in the introduction – had been accepted as routine practice in sewage treatment works.

This situation changed when problems of environmental pollution, especially the pollution of liquid and solid wastes by man-made persistent substances (e.g. heavy metals), got greater importance and the significance of the determination of additional sewage sludge characteristics became evident. It was the time when local authorities and water boards set up regulations for sewage sludge use and disposal and fixed guide values for heavy metals in sludges and soils when sludge was used in agriculture.

In August 1972, the Commission of the European Communities, General Directorate XII started in its Environmental Research Programme the Concerted Action COST 68 "Sewage sludge processing", which had been concluded in Brussels on 23 November 1971 and to which 13 countries contributed during the following two years. The scope of the project was defined as:

> "The assessment of methods of processing and disposing of sewage sludge by the comparative evaluation, using standardized criteria, of existing plants of industrial scale in different countries".

From three study groups, two dealt with problems of sludge characterization and standardization.

The first group, "Standardization", which was coordinated by K.A. Wuhrmann (Switzerland), examined the known sludge parameters if they could be defined and if their determination could be standardized. As result the group drew up a list of recommended parameters for general use and of additional parameters which should be taken into consideration for standardization in the future (see Table 1).

TABLE 1. Cost 68 Sludge Parameters 1974

recommended for general use	considered for further evaluation
Mass	<u>first priority</u>
Densitiy	Centrifugability
Dry residue	Conditioning effects
Undissolved substances	Particle size distribution
Volatile substances	Stability
Non volatile substances	Water binding forces
Calorific value	Heavy metals content
Viscosity (for sludge water)	
Specific resistance to filtration	<u>lower priority</u>
Capillary suction time	Organic pollutants (pesticides, surfactants)
Compressibility	Parasite eggs
Settling rate	Enterobacteria
Coefficient of separation	Viruses
	Zeta- and redox potential

A second group, under the convenorship of L. Ulmgren (Sweden), was set up to work out new methods for sludge characterization or to improve existing methods. The parameters listed in Table 1 considered to be evaluated with first priority were studied in four European laboratories (IVL, Solna/Sweden; WaBoLu,

Berlin/Germany; NIVA, Oslo/Norway and RIZA, Voorburg/The Netherlands) with the objective to draft proposals for further use.

The progress and final results of the project showed a wide span of success some made valuable additional scientific contributions, mainly from EAWAG, Zürich/Switzerland while others encountered certain difficulties during the research work. The results were put together in a final report of the Commission of the European Communities (1975). The work on methods for conditionability and for particle size distribution resulted in draft proposals and respective publications (Leschber and Haacke, 1975; Leschber and Niemitz, 1976). There was no immediate extension of this project although the members of the Management Committee had concluded that the international co-operation had permitted a productive exchange of experience among the participating scientists.

In 1977, after a two years period, the Concerted Action started again but with new title COST 68bis "Treatment and use of sewage sludge" which was scheduled to last three years. This new programme had two extensions, COST 68ter and COST 681, and ended in 1990. During the whole period there were five working parties (WPs) working with experts from 12 European countries and from Canada (guest status) in different fields of sludge technology. Among these groups only one, WP 2 entitled "Chemical pollution of sludge" and named later on "Chemical contamination of organic sludge and soils" dealt with analytical aspects of the determination of heavy metals and organic pollutants and the sampling of sludge and soil .

During this work, progress was made in the determination of the so-called total content of heavy metals by aqua regia digestion and in the evaluation of the mobility of the main heavy metals zinc, cadmium, copper, nickel, lead and chromium. Hereto, four interlaboratory comparisons were performed to which more than fifty laboratories from all over Europe contributed. Nearly the same number of laboratories participated in interlaboratory comparisons on the determination of polychlorinated biphenyls in sludge samples which resulted in a special report of the European Commission (Leschber, Tarradellas, and l'Hermite, 1985) and a later publication (Taradellas, Muntau and Leschber, 1989).

Besides this international co-operation, with the main view to the agricultural use of sludge and pollution problems connected with, the development of methods for the determination of other sludge parameters moved forward in different countries in these years. In England, at the Water Pollution Resaerch Laboratory in Stevenage the characteristics of dewaterability and filtration were elaborated. In France, at the Institut de Recherche Hydrologiques in Nancy, work on physical characteristics of sewage sludge was performed as well as in Italy, at the Istituto di Ricerca sulle Acqua in Bari of the Consiglio Nazionale delle Ricerche.

In the Federal Republic of Germany, a subcommittee "Sludge and sediments" of the Special Group for Water Chemistry (Fachgruppe Wasserchemie) within the German Chemists Society (Gesellschft Deutscher Chemiker) worked from 1969 until 1977 alone and later on in close connection with the German Institute for Standardization (DIN) for DIN standards/Deutsche Einheitsverfahren (DEV) in sludge and sediments. The methods were registered as DIN 38414, group S and comprised in the first phase of work only a small number of standards which were commonly used in wastewater treatment plant monitoring (sampling, determination of water/dry matter content, loss on ignition, pH value, sludge volume/-index).

When pollution aspects (with regard to sludge tipping and disposal and to Sludge Regulations from 1982 for the agricultural use of sludge) gained more and more importance, the catalogue of standards was expanded and the work was intensified. Methods for leaching of sludge, aqua regia digestion as pretreatment for heavy metals determination, chemical oxygen demand as well as the determination of sludge respiration characteristics and the amenability to anaerobic digestion were standardized following the requirements of modern sludge technololgy and disposal.

In the third and current phase the catastrophe of Chernobyl caused activities in the field of radiological methods leading to standards/drafts at the end of the eighties. Soil protection requirements and proper agricultural use of sludge led to methods for the determination of phosphorus compounds, cumulative parameters for organic halogen compounds (AOX, EOX, POX), salmonellae, and recently to DIN draft

standards for the analysis of polychlorinated biphenyls (PCB) and polycyclic aromatic hydrocarbons (PAH). Together with other trace organics, which may possibly be standardized in the future, these methods seem to require a more careful performance of the dry residue determination avoiding loss of volatile organics. This was the reason for including a freeze drying procedure in the subcommittee's programme. Table 2 explains the development of the standardization work described above.

TABLE 2. Standards for Sludge and Sediment Characterization (Din 38414, Pt. 1–23, October 1993)

Part No.	Method	Standard	Draft standard	Draft proposal
1	Sampling of sludges	1986		
2	Moisture content/Total solids residue	1985		
3	Residue/Loss on ignition	1985		
4	Leachability by water	1984		
5	pH value	1981		
6	Sludge respiration	1986	1993 DIN-ISO 8192	
7	Digestion with aqua regia	1983		
8	Amenabilitiy to anaerobic digestion	1985		
9	Chemical oxygen demand	1986		
10	Settled share of sludge volume/ Sludge volume index	1981		
11	Sampling of sediments	1987		
12	Phosphorus compounds	1986		
13	Salmonellae	1992		
14	Specific gross α-activity			1988
15	Residual β-activity	1990		
16	Radionuclides by γ-ray spectrometry		1988	
17	Organic halogenes amenable to stripping/extraction	1989		
18	Adsorbed organic halogens	1989		
19	Water vapour volatile organic acids			1993
20	Polychlorinated biphenyls		1993	
21	Polycyclic aromatic hydrocarbons		1993	
22	Solids residue by freeze drying			1993
23	Ion chromatography of lower fatty acids			1993

PRESENT SITUATION – WORK OF CEN/TC 308

The directive 86/278/ECE of the Commission of the European Communities (1986) set new standards in Europe for the agricultural use of sludge and the handling of pollution problems connected herewith. But although guide/limit values for heavy metals in sewage sludge in this directive were set up, no direct analytical procedures for their determination were given, only a list of parameters to be determined was contained in the annex of the directive. This stood in contrast to e.g. the German Regulations, where detailed procedures were given or respective German DIN standards were cited.

Finally, this lack of investigation on methods and the requirements of modern sludge technology, resulting in increasing amounts of sludge, together with more and more restrictive regulations for sludge use and

disposal, had been the reason for a French proposal to the European Committee for Standardization (CEN) for a new project "Water cycle sludge characterization" in May 1992. After having reached a positive vote among the CEN members and the agreement of the CEN organizational bodies, a Technical Committee, TC 308 "Characterization of sludge", was founded in May 1993. The secretariat and the chairman were given to France and three working groups (WPs) were set up : WP 1 "Characterization methods" (convenor and secretariat Germany), WP 2 "Guidelines for good practice in the production, utilization and for disposal of sludge (convenor and secretariat France) and WP 3 "Measures to preserve, to improve and to extend sludge utilization and disposal routes (convenor and secretariat Great Britain). After the first meeting of TC 308 and an additional meeting of an ad-hoc group for the evaluation of a preliminary programme in 1993 the main aspects of the future work for the standardization of sludge examination methods in an European dimension seem to be the following.

- To develop a method for sludge sampling (work to be done in liaison with ISO/TC 147 "Water quality", Subcommittee 6 "Sampling", according to the Vienna Agreement of CEN and ISO. A respective project had been started by ISO.)

- To elaborate methods quoted in the Directive 86/278/ECE (Using existing methods of water analysis from CEN/TC 230 "Water quality" and ISO/TC 147 if possible). Here CEN standards for the determination of the dry matter content and its organic proportion, the pH value, the nutritive elements nitrogen and phosphorus and/or their compounds, the aqua regia digestion of sludge, and the subsequent determination of the elements cadmium, chromium, copper, lead, mercury, nickel and zinc, will be the first steps.

- To work out methods at the request of TC 308/WG 2 and WG 3. Due to requirements of proper sludge handling, parameters listed in Table 3, besides the ones just mentioned, seem to be the subject of the work of WG 1. The most important ones may be drainability, capillary suction time, specific resistance to filtration, and compressibility as well as centrifugability for the needs of dewatering and thickening of sludge and the leachability for purposes of proper storage or tipping.

At present, an inquiry is being made among the members of TC 308 to know in the near future the priorities for developing CEN sludge investigation methods from the respective national points of view.

TABLE 3. Proposals for CEN/TC 308 Standardization Work (October 1993)

Priority	Physical parameters	Chemical parameters
I/1	Dry residue	pH value
2	Loss on ignition	Aqua regia digestion
3	Capillary suction time	Nitrogen compounds
4	Drainability	Phosphorus compounds
5	Calorific value	-
II/1	Sludge volume/-index	Chemical oxygen demand
2	Specific resistance to filtration	Organic carbon
3	Compressibility	-
4	Centrifugability	-
5	Leachability	-

Comparing Tables 1 and 3 it becomes evident that, with regard to physical parameters for sludge examination, only little progress had taken place in the last two decades. It is hoped that the work of CEN/TC 308 will improve the situation very soon. The European Waste Water Group (EWWG), a body of

the respective technical associations, has pointed out, in its plenary session on 20 September 1993 in a so-called "Position Statement" on CEN/TC 308 work, that EWWG offers its expertise and experience and stresses the urgency of the work which is seen as an adjunct to the legal framework of ECE. EWWG hoped that CEN/TC 308 work will be properly co-ordinated with related similar international activities and will be specifically designed to provide missing technical detail on sewage sludge characterization and methodology (Romano and Thairs, 1993).

REFERENCES

Commission of the European Communities, Secreteriat EUROCOP-COST (1975). Cost Project 68, Sewage sludge processing. *Final Report of the Management Committee. Doc.* **EUCO/SP/48/75-XII/825/75**, 80 pp., Brussels, 20 November 1975.

Commission of the European Communities (1986). Richtlinie des Rates vom 12.Juni 1986 über den Schutz der Umwelt und insbesondere der Böden bei der Verwendung von Klärschlamm in der Landwirtschaft (86/278/EWG). *Amtsblatt der Europäischen Gemeinschaft Nr. L 181/6-L 181/12* vom 4.7.1986.

Leschber, R. and Haacke, W. (1975). Klärschlammuntersuchung unter besonderer Berücksichtigung der Teilchengrößenverteilung. *Vom Wasser* **45**, 305-325.

Leschber, R. and Niemitz, W. (1976). Erarbeitung von Methoden zur Ermittlung der Konditionierbarkeit von Klärschlämmen. *Vom Wasser* **47**, 187-207.

Leschber, R., Tarradellas, J. and l'Hermite, P. (Editors, 1985). Polychlorinated biphenyls (PCB). Determination in sewage sludge and related samples. Results of an interlaboratory comparison. *Commission of the European Communities Doc.* **SL/111/85**, 96 pp., Brussels

Romano, P and Thairs, T. (1993). EWWG Position Statement on CEN Technical Committee 308: Characterization of sewage sludge. *Draft for the European Waste Water Group plenary session on 20 September 1993*, 2pp.

Taradellas, J., Muntau, H. and Leschber, R. (1989). Interlaboratory comparisons on the determination of PCBs as a model case for organic substances in sludge. In : *Organic Contaminants in Wastewater, Sludge and Sediment : Occurrence, Fate and Disposal* (Editors: D. Quaghebeur, I. Temmerman and G. Angeletti), 81-93. Elsevier Applied Science, London/New York.