Gonzalez, Annals of Environmental Science / 2012, Vol 6, 101-122

A RETROSPECTIVE ASSESSMENT OF ARTIFICIAL, FORMULATED AND SYNTHETIC SEDIMENT

Adrian M. Gonzalez*

Department of Civil and Environmental Engineering,
University of Tennessee, Knoxville, TN 37996, USA

Received June 20, 2012; accepted November 16, 2012

ABSTRACT

Understanding aquatic sediment's inherent complexity requires multiple assessment tools and lines of evidence to explain its composition, character and function. Data quality of sediment assessments can suffer from a lack of suitable "control" or "reference" sediment material; natural sediment is too dynamic and heterogeneous to serve this role. One promising solution is to create surrogate sediment substrates (3S) composed of synthetic materials. The development of 3S has not progressed beyond a few decades-old approaches. Taking inventory of current conditions often can clarify context and goals, and inspire new ideas to overcome the current stagnation. To this end, the author reviewed 114 articles that report using 3S in studies of sediment mechanics, sediment ecology (habitat), and toxicity and bio-assimilation of sediment-associated chemicals. The review disclosed an apparent systematic error in previous synthetic sediment formulations: the presumed silt components were actually clay minerals, perhaps leading to misinterpretation of results under invalid composition assumptions. Organic matter composition and other higher-level sediment characteristics continue to be major challenges to progress. The review identified a few solitary efforts at incorporating high-level sediment properties in 3S formulations, such as inherent nutritional value of synthetic sediment, acid-volatile sulfide content, oxidation-reduction potential, reproducible conditioning and equilibration, and microbiological populations. It also revealed an underlying presumption that a single (currently elusive) universal formulation can meet all sediment study objectives. This unspoken presumption has imposed unnecessarily daunting expectations. An alternative proposal is to develop multiple 3S materials, each formulated with assessment- or DQO-specific characteristics pertinent to their intended use. The information consolidated in this review can be a platform for future progress in the field.

Keywords: synthetic sediment, sediment data quality, reference sediment, control, formulation

1. INTRODUCTION

Environmental media are not mutually isolated entities; chemical stressors in one (e.g., surface water or groundwater) can affect the condition of others (e.g., aquatic sediment) [1,2]. As a result, sediment quality management (SQM) has become an important component of many industrialized nations' environmental policy framework [3-8]. Aquatic sediment serves multiple functions simultaneously. Among the more obvious are 1) a dynamic geologic formation created by deposition of materials from up-gradient sources; 2) a habitat structure for aquatic flora and fauna; 3) a link in the biosphere's energy flow continuum; 4) a substrate that interacts with chemical stressors. Sediment is influenced by external environmental conditions, and is composed of numerous factors that are inter-related through highly complex processes and relationships [9]. Understanding its inherent complexity requires a comprehensive approach, including deconstruction and detailed analysis of individual factors ("reductionist") and integration of advances in multiple fields ("holistic") [9]. The former generates and tests hypotheses for details of the aquatic sediment composition, character and function (CCF); the latter serves as a check on intra- and inter-consistency of results across multiple fields of study.

SQM has matured as a discipline over time in parallel with expanding regulatory oversight of the environment, advances in engineered mitigation techniques, and improvements in scientific tools available to researchers and managers of sediment quality [10-12]. There is no indication that this trend will change substantially in the foreseeable future; advances in SQM and sediment science are needed for many currently unanswered questions, and will be needed for currently undisclosed problems [8]. Collectively, sediment-specific assessment tools (both reductionist and holistic) provide lines of evidence for describing sediment CCF. The weight of these lines of

---

* Corresponding author email: envonyx@gmail.com, phone: 865-673-4820, fax: 530-689-8330

evidence depends on factors such as the representativeness of sediment samples of conditions in the broader environment, sampling density (either spatial or temporal) to adequately capture a range of conditions at the sampled location(s), and quality and reliability of data obtained from sediment-specific analytical tools [13]. Obtaining high quality data from aquatic sediment systems can be challenging because of the complexity of its CCF; the inherently large number of components, processes and functions makes it an arduous environmental medium to characterize with high levels of precision. Thus, these two objectives, understanding sediment substrate complexity and precise control of study variables, appear to be intractable with currently available tools. New tools are needed if we are to make progress in providing greater control of study variables to aquatic sediment scientists or sediment quality managers.

Current sediment quality management suffers from limitations in data quality, specifically by the lack of a suitable “control” or “reference” sediment material for some sediment assessments. One promising solution to this dilemma is the use of entirely synthetic materials. Surrogate sediment substrates (3S) (i.e., artificial sediment, formulated sediment, synthetic sediment) have been proposed as potentially useful tools for sediment studies; however, current guidances for their use are essentially conceptual descriptions of how the concept can improve sediment data quality [4,12,14,15]. If this concept is to reach its full potential there needs to be a shift from generalities to specific details. To this end, the literature was reviewed for information on previous work with 3S materials. The value of this review is in compiling a comprehensive overview of 3S development to date and perhaps inspiring new ideas and renewed interest in the topic.

2. LITERATURE SURVEY AND OVERVIEW

The author has compiled a database of over 2600 articles (inclusive of journal papers, reports, dissertations, publications, etc.) associated with sediment science and related fields of study. From that database, 114 articles, having direct relevance to 3S materials were culled and evaluated [16-129]. These 114 articles were published over a 46-year period, authored or co-authored by some 244 individuals from dozens of countries or provinces, and published in nearly 100 journals, reports, governmental agency guidance documents, or conference proceedings. Other published works on the topic may exist and may have been overlooked by the author; however, these 114 articles are a robust representation of the history and status of 3S development.

The 114 articles [16-129] were organized chronologically and by study category (Table 1). The period covered by the time-line starts at 1964, the earliest known example of the use of surrogate particles for studying natural aquatic sediment. The numbers in the main body of Table 1 correspond to the references listed at the end of this paper.

Three categories of references are displayed in Table 1: numbers in parentheses correspond to references that report new ***procedures*** for creating synthetic sediment materials; underlined numbers correspond to references that report on ancillary concepts or methods that this author judged will be useful to future 3S development work (discussed later in this article); and numbers with no special formatting correspond to references that report the ***application*** of surrogate materials that were created using existing 3S protocols; new procedures are not presented in those references.

Numbers in the far right column and near the bottom of Table 1 represent count totals: the number of studies in each Study Category (in the far right column) and total number of studies published in the indicated year (at the bottom of each column). The two rows at the very bottom of Table 1, below the main time-line area, identify the publication years of either work on synthetic soil (indicated by check marks) or governmental guidance documents that suggest the use of 3S materials (indicated by capital letters). The information in the two bottom-most rows is included to provide additional context for understanding the history of 3S materials.

The scope and study objectives of most of these 114 articles were found to belong to one of four general categories defined later: 1) studies of physical processes in the environment; 2) studies of ecological functions or processes of specific aquatic organisms (independent of chemical stressors); 3) toxicological studies of the biological effects of chemical stressors on specific aquatic organisms; and 4) “bio-assimilation” studies of uptake of chemical mass into organism tissues. A miscellaneous category is included to capture the few studies that could not be assigned to any of the other four. Articles in three of the categories (ecology, toxicity and bio-assimilation) were further delineated by organism class investigated aquatic macro-flora (e.g., submerged or emergent plants in wetland or marsh environments), aquatic macro-invertebrates (benthic or epibenthic), and microbial species (flora or fauna: bacteria, algae, etc.).

Table 1 Chronology of 3S[a] Development

	Year Published (Decade & Last Digit of the Year): Table entries correspond to References Listed at End of Paper																																					Totals[c,d] by Study Category
	Pre-1980				1980s										1990s										2000s										2010s			
Study Category[b]	1964	1976	1978	1979	0	1	2	3	4	5	6	7	8	9	0	1	2	3	4	5	6	7	8	9	0	1	2	3	4	5	6	7	8	9	0	1	2	
Physical Processes (e.g., sorption)	16		17				20	22				26		28							52 56	61 63	67	69 74	78 80		88 90 91	94		102 107 109	110 111		116 117 119 120		126			29
Ecology of Flora (e.g., wetlands)				18	19			21							(29)	(33) 35 36	(37)																					8
Ecology of MACRO-Invertebrates									24				27		(31)	(34)	(37) (38) (39)		(42)		51	60		72	76		92	93 95	96	105								17
Ecology of Microbial Invertebrates															30						58		66	73	78 79		89		98	104 108								10
Toxicity to Flora (e.g., wetlands)																(32)				47			65															3
Toxicity to MACRO-Invertebrates																			40 (41) 43 (44)	45 46 (48)	49 50 53 54 57	59	64 65 68	70 (71) 72 (75)	77	(81) 82 83 84 87		94	96 97 99 100 101	106		114 115		123	(125) 128			38
Toxicity to Microbial Invertebrates																							65															1
Bio-assimilation[c] in Flora (e.g., wetlands)																															112							1
Bio-assimilation[c] in MACRO-Invertebrates								23		25												59 61 62		74	76 80	83 85 86	88			103 107	110	113	116 121	124	126 127	129		22
Bio-assimilation[c] in Microbial Invertebrates							20	22																														2
Miscellaneous Studies																					55												118 122					3
Annual Study Totals[d]	1		1	1	1		2	4	1	1		1	1	1	3	5	4		5	4	10	7	7	9	8	8	6	4	7	9	4	3	8	2	5	1		134
3S Development Work[f,g]															2	3	3		3	1	1		1	4	1	2	2	1	1	2			1		1			29
Synthetic Soil work		√		√	√				√					√				√																				
Guidance Documents[e]									A										B	C		D			E				F						G,H			

a) 3S = surrogate sediment substrates (any combination of materials intended to represent or simulate the composition, characteristic(s) and/or function(s) [CCF] of natural aquatic sediment); b) Numbers in the timeline-table correspond to numbered references listed at the end this paper. A paper can appear in a column more than once, if judged to apply to multiple Study Categories; c) Bio-assimilation is a collective term for any of the following processes: bioconcentration (water-to-organism), bioaccumulation (diet-to-organism) or biomagnification (organism-to-organism) pathways; d) Totals correspond to total number of instances, not total number of citations [see Note (b)], and do not include references reporting synthetic SOIL work; e) Letters correspond to the following reference or guidance documents: A - OECD, 1984 (No. 207); B - USEPA, 1994 (EPA 600/R-94/025); C - CCME, 1995 (No. EPC-98E); D - CCME, 1997 (Tissue Residue Guidelines); E - USEPA, 2000 (EPA/600-R-99-064); F - OECD, 2004 (No.218); G - ASTM, 2010 (Method E 1706-05); H - OECD, 2010 (No.233); f) Values in parentheses correspond to references with a primary focus on developing 3S materials; g) Underlined values correspond to references that report a procedure or concept that can be used to improve 3S materials.

As mentioned earlier, some articles contain results that pertain to multiple study objectives, or yielded results pertinent to more than one hypothesis. In these few cases, the article's reference number is listed in Table 1 multiple times under its publication year column, once in each pertinent Study Category row. Thus, there are 134 study objectives addressed in the 114 articles culled from the database.

Research addressing physical processes (29 total instances) report on geo-physical factors related to topics such as stream bed mechanics, influence of wave energy on sediment mobility, or the interactions between chemicals and particle-surfaces (i.e., partitioning/absorption, adsorption, desorption, hysteresis effects, etc.). Studies addressing "ecology of" topics (35 total instances) focus on physical or geochemical factors in a substrate that influence an organism's performance (survival, growth, reproduction, etc.) when exposed to that substrate. They consist of lab-based studies of "sediment habitat" requirements for culturing benthic and epibenthic organisms, as well as studies of sediment factors affecting species' performance in the field. Chemical stressors were not included in the study designs of "ecological function" studies. They were included in studies in the "toxicity" category (42 total instances), which evaluated biological response(s) of organisms exposed to chemical stressors, at any level of injury: acute, chronic, sub-chronic, systemic damage, etc. The fourth category is "bio-assimilation" (25 total instances), a term used in this paper to identify any of the processes by which organisms acquire increased body burdens of chemical stressors: bioconcentration (water to organism), bioaccumulation (diet to organism) and biomagnification (trophic-level to trophic-level).

2.1. The Beginning: Pre-1990

Few pre-1990 articles were found that reported studies using any type of 3S material, and no pre-1990 articles were found whose primary objective was to create realistic synthetic sediment (Table 1). The 13 identified pre-1990 articles represent relatively isolated instances of 3S use, with no coordinated or long-term intention of continued or expanded use of the concept. The studies reported in these 13 articles used surrogate particles (singly or in simple mixtures) for the following objectives:

- Evaluate particle responses to physical perturbations [16,17,22,26,28];
- Serve as support and nutrient release substrate for macrophyte root-systems [18,19,21] or microbial growth [20,22];
- Serve as an inert substrate in studies of pesticide bioconcentration from spiked water [23,25]; or
- Evaluate particle ingestion processing of benthic invertebrates [24,27].

One notable study [20] used a mixture of potting soil and clay as a surrogate sediment material to study its interaction between bacteria and metal contaminants. This article is the earliest direct attempt at simulating natural sediment; their simple soil/clay mixture did not achieve a good degree of realism to natural sediment.

2.2. The Early Period: 1990 – 1992

Use of 3S materials in environmental studies began in earnest after 1990 (Table 1), partly in response to maturing environmental policies in industrialized nations [2]. In the years leading up to 1990, a number of foundational environmental laws were enacted in the U.S.: the Great Lakes Water Quality Agreement (1972), Clean Water Act and amendments (1972, 1977, 1987), Toxic Substances Control Act (1976), the Resource Conservation and Recovery Act (1976), the Comprehensive Environmental Response, Compensation and Liability Act (1980) and its reauthorization in 1986, and the Ocean Dumping Ban Act (1988). This legislative activity is reflected in Table 1 by capital letters in the "Guidance Documents" row along the bottom of Table 1; each letter represents a governmental agency guidance document published in the indicated year (see Note (e) in Table 1). Compliance with these regulatory requirements created the need to evaluate environmental media, including aquatic sediment. The number of published articles involving some form of 3S material increased from 13 disparate publications in all years prior to 1990, to over 50 in the 1990 decade alone, and the specificity of study objectives in those articles expanded and deepened as well (Table 1).

Development of surrogate soil material by soil scientists likely inspired the idea of creating uncontaminated synthetic aquatic sediment. Workers from Corvallis, Oregon created microcosm chambers containing synthetic soil and used them to study the distribution and fate of pesticides [130], fungicides [131] and herbicides [132] in plants, water, invertebrates and voles. The soil recipes in their studies were the forerunner of synthetic soil formulations recommended in government guidance documents [133-135] and the scientific literature [136-139]. Those synthetic soil formulations, in turn, were the basis for many synthetic sediment formulations (e.g., [97] and [128]).

The three years between 1990 and 1992 were marked by concerted efforts to create surrogate aquatic sediment. Several papers from that time period report work associated with wetland ecology and toxicology studies by Walsh, Weber and associates at USEPA in Florida [29,32,33,37] and the pioneering hexagenia culturing work by Ciborowski, Corkum, Hanes and others at the Ontario Ministry of the Environment in Canada [31,34,38,39]. This productive 3-year period generated 12 articles describing the creation and use of synthetic sediment, eight of which were specifically focused on reporting components and procedures for creating such materials (Table 1). These two groups of investigators were responsible for all activity related to using or developing 3S materials in the aquatic sciences during this early period. Their work served as the foundation for most 3S work done since then.

2.3. Recent Work: 1994 – 2011

The 1993/1994 period marked the beginning of a notable expansion in the number and scope of studies involving 3S materials. The number of articles reporting studies using 3S materials increased in almost every Study Category (Table 1). The following statistics compare numbers of published 3S-related articles before and after 1993:

- Physical processes: 6 before / 23 after
- Ecology of aquatic flora or fauna: 16 before / 19 after
- Ecotoxicology (effects of chemicals on aquatic biota): 1 before / 40 after
- Bio-assimilation: 4 before / 21 after (representing all bioaccumulation, bioconcentration and biomagnification processes)

Activity in the “ecology of” Study Category (using 3S materials in ecological function studies) moderated after 1993; almost as many articles dealing with infaunal ecology were published between 1994 and 2005 as were published in the brief but productive 1990-1992 period (Table 1). The more striking (but not surprising) observation is the sudden and simultaneous publication of articles reporting 3S-related studies of toxicity to aquatic/benthic organisms, physicochemical mechanisms between contaminants and abiotic sediment components, and bio-assimilation of toxicants into aquatic/benthic organisms (Table 1). These three fields of study are inter-related, and the sudden popularity of applying 3S materials reported in those studies undoubtedly reflects deepening interest of sediment quality managers in gaining better understanding of the chemical-sediment-benthos relationship, as well as availability of sophisticated experimental techniques and analytical instrumentation.

The production rate of studies incorporating 3S materials has been steady to slightly declining since 1994. Use of 3S materials in benthic and epibenthic ecology studies appears to have lost its value or popularity; the last published work in this category found by the author was in 2005 (Table 1). No articles were found from the post-1993 time period that used 3S material to study ecological function(s) of aquatic or semi-aquatic macrophytes (e.g., wetland or marsh flora) or bio-assimilation of contaminants by microbiological species (Table 1). Only single publications were found addressing: chemical toxicity to rooted/vascular aquatic plants [47]; chemical toxicity to phytoplankton and bacteria [65]; the response of the Microtox® chemilluminescent bacterium to commercially available contaminated reference sediment [118]; and bio-assimilation of a pesticide by the common cattail [112].

Most published 3S work since 1993 has focused on physicochemical processes between chemical contaminants and physical component(s) of 3S material (23 articles), ecology of macro-invertebrate species (10 articles) and micro-invertebrate species (9 articles), toxicity of contaminants to benthic macro-invertebrates (37 articles), and bio-assimilation of contaminants by benthic macro-invertebrates (20 articles). Clearly, since 1994 the most popular (i.e., well-funded) areas of research that incorporate 3S materials are macro-invertebrate toxicity, macro-invertebrate bio-assimilation, and understanding the fate of contaminants in the aquatic sediment compartment (Table 1).

3. STANDARD TERMINOLOGY

The literature review exposed inconsistent use of terminology related to 3S development. The terms “formulated,” “artificial,” “synthetic,” “reference,” “control” and “amended” have been used interchangeably and loosely; “formulated” and “artificial” are the most commonly used terms to identify 3S material. In the spirit of scientific rigor, the author proposes standardizing the definitions of these terms, as follows:

Let **reference sediment** refer to field-collected, presumably uncontaminated, natural sediment used without modification (except perhaps for minor alteration such as manually removing stones, twigs, and other bulk objects). The quality of reference sediment depends entirely on all known and unknown environmental processes that may have acted on the sediment at that field location.

Let **amended sediment** describe any field-collected natural sediment that is later amended with one or more extraneous materials. Presumably, any such modification would be implemented so as to meet a specific study objective. The quality of amended sediment depends on the CCF of the field-collected material and of extraneous material(s) added to it.

Reserve the term **artificial sediment** for substrates comprised of simple homogeneous materials (e.g., sand, glass beads, silica gel, shredded paper towels, clay, etc.). It would be understood that “artificial” material is never intended to simulate the CCF of natural whole sediment. Rather, these individual homogeneous materials serve very specific study objectives; e.g., to serve as an innocuous solid substrate in an experiment or to represent one specific component of natural sediment. The quality of artificial sediment depends on the CCF of the homogenous material used as substrate, which often can be documented with product specification sheets provided by suppliers.

Synthetic sediment should be the standard term for a composite 3S material, prepared (i.e., *synthesized*) by combining more than two homogenous materials (i.e., “surrogate components”) with the singular objective of simulating natural aquatic sediment. Each component material used in synthetic sediment should represent one or more natural sediment components, and can consist of a single homogenous material (e.g., commercially available materials such as sand, silt, clay, a mineral, a pure chemical, etc.), or of an intermediate composite material. An intermediate composite material is a single material prepared from two or more simple homogenous materials (e.g., silica gel particles coated with hematite; glass beads coated with protein). The quality of synthetic sediment depends on the CCF of all components included in the composition, as well as protocol-specific quality objectives for preparation methods.

The terms **surrogate sediment** or **surrogate sediment substrate** (3S) are proposed as broad generic terms for prepared or amended materials (i.e., non-native) intended for use in sediment studies. Three of the four categories defined previously (amended, artificial and synthetic) should be thought of as subsets of the 3S category. Reference sediment, as defined in this paper, is natural, unmodified field-collected sediment, and thus does not belong in the 3S category.

The word **formulated** in the literature is used most commonly as an adjective (e.g., “formulated sediment”); however, that particular usage can be ambiguous. *Formulated* could describe a natural sediment sample that was amended, a particulate material that was coated with another substance, or a true synthetic material comprised of several individual components. To avoid ambiguity, the word “formulated” should be reserved for use as a verb describing the *process* by which 3S material is prepared (e.g., “synthetic sediment is formulated from uncontaminated components”). The word “formulation” can be used as a noun synonymous with *recipe* (“The synthetic sediment formulation accurately replicated the natural sediment sample’s properties”).

4. LITERATURE CONTENT

The various substrates documented in the 3S literature classify into one or more of the four defined 3S types (reference, amended, artificial or synthetic). Several articles report using substrates from more than one of these categories; thus, there are 159 reported uses of 3S substrates among the 114 articles [16-129].

4.1. Reference Sediment (REF)

The author’s literature database contained 37 references to the use of field-collected natural sediment as reference material; many more probably exist in the literature at large. No particular sediment type was identified as more likely to be “pristine” (free of contamination) or more suitable as reference sediment. With very few exceptions (e.g., [140]), reference sediment was qualified as acceptable by either assumption or direct history/knowledge of the location (i.e., direct analysis, distance from any known source of pollution, prior history of use as reference material; etc.).

The natural sediment samples used in these studies apparently met the study-specific objectives (e.g., acceptable organism survival in control treatments using reference sediment; [140]). Using superficially or infrequently characterized natural sediment as reference material carries some risk of it being a source of error in studies. Obvious sources of error include uncertainty in the materials’ history or total contaminant load, the inherent variability in sediment CCF, and potentially limited availability from one collection location or season to the next. Synthetically replicating the CCF of field-sampled reference sediment was not a study objective in any of the 37 references.

4.2. Amended Sediment (AMD)

Nine references to using modified natural sediment were found in the 3S literature. The most frequent reason for modifying the original sediment was to study the influence of the altered characteristic on some study endpoint (e.g., toxicity, partitioning capacity or strength, bioaccumulation, etc.). Some modifications included adding extraneous material to the field-collected sediment (e.g., increasing the proportion of organic matter in the sediment), while others physically altered the natural sediment in some way (e.g., homogenizing or sieving or drying). The amended sediment used in these 9 studies apparently met the study-specific performance-based objectives; however, the error potential and uncertainty in CCF of natural field-collected sediment also applies to amended sediment.

4.3. Artificial Sediment (ART)

A number of studies (44) required a physical substrate on which to create the experimental conditions. In these studies, homogeneous single-component or two-component solid materials included sand, glass beads, metal oxides and silica gel. Their purpose was not to replicate characteristics of natural sediment; they served various purposes, for example as an aquatic plant growth medium, a benthic invertebrate culture substrate, a particle-surface on which humic substances were coated, a homogenous material to study mechanical or physical perturbations in streams, etc. The purity of these relatively simple materials was controlled or well-characterized.

4.4. Synthetic Sediment (SYN)

The author identified 70 references to developing new, or using existing, synthetic sediment; 66 report original peer-reviewed research and four are government agency guidance documents that recommend use of synthetic sediment. The study objectives reflected in the 66 research articles classified into one of two categories: developing or demonstrating new formulations or recipes (the "developer group"; 30 papers), or using existing formulations to answer toxicological or ecological questions (the "user group"; 36). In all cases, the selected formulation was prepared specifically for simulating natural whole sediment; those in the user group relied on the work of the developer group.

4.5. Synthetic Component Materials

The 114 references reporting use of artificial (44) or synthetic (70) sediment represent a large diversity of methods and study objectives. It seemed instructive to list the materials used in those studies. The list of materials in Table 2 is divided into mineral phase materials and organic phase materials. The entries are listed roughly in order of frequency used among these studies, but an entry's position within Table 2 has no other significance.

The 31 materials listed under "Mineral Phase Components" include seven varieties of sand and eight varieties of clay. Although most of the varieties may be chemically equivalent (e.g., most sand varieties may be silica-based), they are transcribed exactly as shown in the corresponding article. Doing so reveals the variety of sources used by 3S users to date. Apparently, the oft cited admonition to standardize materials and sources used to prepare synthetic sediment (e.g., [4,12,14,15]) has not been followed to date. The reviewed articles implied that convenience, familiarity and local availability dictated use of a particular source of mineral component. There was no explicit documentation of attempt to acquire mineral (abiotic) materials from one specific source in the articles reviewed.

Despite the wide range of sources of mineral components reflected in the literature to date, no serious source-dependent issues with the 3S materials were apparent from this review. This casts doubt on the criticality of using materials from a single universal source. In fact, one article [138] reported the need to study quality differences in organic matter from various sources because the investigator's original potting soil source was no longer available. This weakens the argument for single universal sources for individual components. It is counter-productive to constrain synthetic sediment development with a universal source requirement. A practicable alternative is to use "performance criteria" to demonstrate and document the quality of synthetic sediment components. Component function is more important than source.

The organic phase entries in Table 2 consist of 23 individual materials, 16 of which are complex, uncharacterized mixtures of natural organic matter (e.g., "humus"; lamb manure; etc.).

Table 2 Materials Used as Surrogate Substrates (Years: 1964-2012)

Mineral Phase Components	Organic Phase Components
Sand - quartz, play, industrial, foundry	Natural soil - topsoil, potting soil
Sand - sterilized, marine, silica (sil-co-sil)	Natural algae, Spirulin
Clay - kaolin, bentonite, illite, chlorite	"Leafy shoots"
Clay - DMDA-substituted montmorillonite	Leaves - maple, stinging nettle
Clay - potter's, sculptors', modeling	BSA (bovine serum albumin)
Calcite, natural chalk	Peat humus (commercial; dried)
Dolomite	Sphagnum peat moss
Sulfur	Compost - cow manure, lamb manure
Mineral Oxides - aluminum, manganese	Humic acid (Aldrich), particle-coated
Iron oxides - Fe_2O_3, Fe_3O_4	Fulvic acid
Titanium oxide (anatase)	Humus (commercial)
Glass beads	Bran cereal
Commercial resin - Dowex 50W	Commercial - Preparative C-18
Commercial resin – 1 x 8-400	Alpha-cellulose
Commercial resin - SP-650M, phenyl 650M	TetraMin flakes (fish food)
Metal sulfides - iron, manganese	Meat extracts
	Cholesterol

Use of such material is an attempt to simulate sediment organic matter heterogeneity; however, their representative-ness of actual sediment organic matter, or their source/quality consistency, is questionable. As with the mineral phase, the organic phase suffers from a lack of consistent or standardized source. Complying with the “single source” constraint is even more daunting for the organic matter component. Natural sources of organic material are subject to numerous natural processes that can affect the quality of the material from batch to batch (e.g., “manure” from the same animal might change over time due to dietary and other natural physiological changes). Using simpler materials (e.g., alpha-cellulose or humic acid) gives greater control of composition and quality, but it reduces the degree of realism in terms of heterogeneity and complexity.

5. DEVELOPMENT OF SYNTHETIC SEDIMENT

As mentioned earlier, 36 of the 66 synthetic sediment articles report using existing formulations in their studies (the “user” group) (Table 1). The other 30 of the 66 articles (the “developer” group) report new recipes (16) or evaluate the limitations of existing formulations (14). These 30 articles represent the core synthetic sediment development literature to date. They are discussed in the following sections.

5.1. Materials and Methods of Synthetic Sediment Formulation

The 16 articles that reported a new synthetic sediment recipe were reviewed in greater detail to extract information on compositions and components. Compositions and component properties were compared and contrasted, with the objective of identifying relationships among the current synthetic sediment formulations. Any underlying presumptions or patterns in thinking should be revealed by this analysis. The results are compiled in Table 3.

Each composition is associated with a literature article represented at the top of the table by its assigned reference number (and year of publication). Formulation components are listed in the left column (within subcategories: sand phase, silt phase, clay phase, organic phase and other phase). Also included is a count of instances that a particular component was used (under the “Use Freq[uency]” column). For reference, the right-most column (“OECD Soil”) indicates components of synthetic soil used for terrestrial earthworm toxicity tests [133-135].

Table 3 Compositions of Synthetic Sediment Formulations

Component	Use Freq	Reference Number and Year Published																OECD Soil [1]
		29	31	32	33	34	37	38	39	41	42	44	48	71	75	81	125	
		1990	1990	1991	1991	1991	1992	1992	1992	1994	1994	1994	1995	1999	1999	2001	2010	
SAND Phase																		
Silica (2000-500 µm)	6	x		x	x		x			x	x							x
Silica (250 - 50 µm)	7	x		x	x			x	x	x	x							x
Silica (500 - 250 µm)	6	x		x	x	x				x	x							x
Play sand (limestone based?)	1											x						
"Sand (acid washed)"	1												x					
Quartz (White quartz No.1)	2													x			x	
Calcinated sea sand (400-100 µm)	1														x			
Calcitic Sand (industrial; 50-190 µm)	1															x		
SILT Phase																		-
Attacote (attapulgite clay > 2µm)	3	x		x	x													
ASP400 (kaolin-based clay > 2µm)	7	x		x	x		x			x	x			x				
CLAY Phase																		
Attagel (attapulgite-based)	3	x		x	x													
Attasorb LVM (attapulgite-based)	3	x		x	x													
"Potter's" Clay[2]	2		x			x												
"Sculptor's" Clay[2]	2							x	x									
ASP900 (kaolin-base)	2									x	x							x
ASP600 (kaolin-base)	5									x	x		x		x		x	x
Montmorillonite	3									x	x					x		
"Artist's" Clay [2]	1											x						
Kaolinite Clay	1															x		
Chlorite Clay	1															x		
ORGANIC MATTER Phase																		
Peat humus (commercial; dried)	2	x		x														
Sphagnum moss peat	5				x		x						x			x	x	x
Cow manure/compost	2				x		x											
Humic acid	2				x		x											
Potting soil	5		x			x		x	x			x						
Organic humus	3									x	x	x						
Bran cereal (ground fine)	1											x						
alpha-Cellulose	3													x	x		x	
Humic acid coating on clay	1														x			
Cellulose fibers	1																x	
OTHER Phase																		
Dolomite	6				x		x			x	x			x		x		
Food supplements provided	4									x	x		x		x			
Calcite ($CaCO_3$)	3												x		x		x	x
Aluminum oxide (Al_2O_3)	1															x		
Iron (III) oxide (Fe_2O_3)	1															x		

1 Organisation of Economic Cooperation and Development (OECD) Soil Testing Guidelines: No. 207 [Reference 133], No. 222 [Reference 134] and No. 317 [Reference 135]; 2 Unknown composition.

Table 3 does not identify the *only* instances of the use of these particular components; it identifies what is believed to be the *first* identified use of these components in studies that claim to report a new synthetic sediment recipe. Studies in the "user" group of articles also used one or more of these same components.

The four general categories in Table 3 are universally invoked when sediment composition and quality are discussed. Most associate the terms sand, silt and clay with relative particle size, not with geochemical composition. In most cases, this imprecision is not important; however, simulating natural sediment with synthetic materials to a greater level of realism and specificity will require careful definition of terms and rigorous attention to detail. The silt phase category in Table 3 provides a clear and relevant example of this need. A quick search on the geochemical composition of the two "silt" components, "attacote" and "ASP400," reveals that both are compositionally and structurally clay minerals. Attacote is a commercial name for *attapulgite*, a non-swelling mineral hybrid of smectite and palygorskite clays, consisting of magnesium aluminum phyllosilicate in a three-dimensional, needle-like structure [141]. ASP® products (ASP®400, ASP®600 and ASP®900) are marketing labels for a material composed of impure *kaolin* clay (ASP® is derived from "Aluminum Silicate Particle") [142]. In fact, the specification sheet for ASP®400 directly states that the 90th percentile particle size (D90; 90 percent of particles are smaller than the given value) is 2 microns [143].

The significance of this is that synthetic sediment formulations reported in the literature appear to have lacked a true silt component, and instead contained a greater proportion of clay than the preparers intended or knew. Their experimental results have been characteristic of essentially *sand-clay* substrates and not of sand-silt-clay substrates as they presumed. The systematic error from underestimating the clay content and overestimate the silt content is not trivial. Calling clay mineral "silt" because its median particle size is above the operationally defined 2-µm silt-clay margin ignores the unique geochemical properties associated with true silt mineralogy, and could lead to distorted conclusions regarding synthetic sediment functionality. Unknowingly, this may have hindered substantive progress in developing compositionally and functionally realistic synthetic sediment material. This is remotely analogous to the colloid-dissolved issue, which to some degree is complicated by the *operationally* defined boundary at 0.45 µm (e.g., [144]).

A substantial proportion of natural silt particles has a composition that is geochemically closer to sand, (i.e., non-clay minerals quartz, oxides, carbonates, feldspar, and mica, among others), than to clays [145]. The geochemical composition of particles has a profound influence on their behavior and function in natural settings [146, 147]; the fundamentals of sedimentation and sediment transport modeling derive in large part from particle composition and the resulting surface properties [148, 149]. Simulating natural sediment with greater realism and specificity must incorporate distinctions of this type.

The data in Table 3 indicate a slight temporal trend in component selection. The time frame represented can be divided into two periods demarcated at about 1994. There was very little variation in the materials used to represent sand and pseudo-silt phases; the clay phase was composed of essentially one of three types of clay mineral: montmorillonite/bentonite, kaolin or attapulgite. Most variation in composition was in the organic matter phase, reflecting some trial-and-error exploration. The brief but productive 3-year period (1990-1992) yielded formulations with practically identical components. Formulations after 1994 were created with a few more components: sands were still predominantly silica-based, the pseudo-silt component was still based on ASP, but a few supplements (dolomite, calcite or food material) began to be added to formulations. Some studies used sophisticated, composite materials (e.g., humic acid coated clay) to represent the sediment organic matter phase.

5.2. Limitations of Current Formulations

In addition to the 16 articles reporting methods and materials for preparing synthetic sediment, another 14 articles reported on auxiliary topics or concepts relevant to aquatic sediment. Those studies demonstrate how current formulations are deficient in simulating many characteristics of natural sediment such as diversity of organic matter quality or type, nutritional value of synthetic sediment to benthic organisms, acid-volatile sulfide, equilibration conditions and time, or the microbiological aspects of natural sediment. The implied conclusion in each case is: Because **current** synthetic sediment formulations do not simulate these higher-level characteristics, their degree of realism is low and therefore their usefulness to sediment assessment is limited.

5.2.1. Factor: Organic Matter Character (quality and type)

It had been suspected early on that the effectiveness of synthetic sediment to serve as a standard reference material was directly related to its degree of realism, and that accurately simulating natural sediment is not a trivial exercise. This hypothesis has been tested experimentally by several investigators. Fleming, Holmes and Nixon [68] prepared several synthetic formulations with varying organic carbon concentrations and clay content equal to that of a sample of natural sediment, but using two different types of organic matter (peat versus alpha-cellulose). Besser et al. [94] studied the fate, bioavailability and toxic effects of metal contaminants in numerous synthetic sediment formulations having varying concentrations and types of organic matter. Authors of a third study [119] synthesized a humic acid-mineral particle complex and used it to study the effect of the organic coating on the fate of uranium ion in the system. All of these studies clearly demonstrated the importance of organic matter quality (type): they showed marked differences in contaminant sorption/ binding to sediment or in contaminant bioavailability to macro-invertebrate test species. The quality (type) of organic matter profoundly influences the degree of realism achieved by synthetic sediment formulations.

5.2.2. Factor: Nutritional Requirements from Substrate versus External Supplements

A corollary of the organic matter quality factor is the question of whether synthetic sediment can be formulated with organic matter that has inherent nutritional value to test invertebrates, or if exogenous nutritional supplements will always be required. A study by Lacey, Watzin and McIntosh [70] exposed benthic invertebrates to multiple uncontaminated synthetic sediment batches prepared using one of three types of organic matter (peat, alpha-cellulose or maple leaves) and at varying concentrations, and observed that their survivorship varied widely relative to the organic matter type and concentrations in the substrate samples. A Master's thesis by Wastlund [72] addressed the same question and showed that different organic matter types incorporated into synthetic sediment formulations greatly influenced the test invertebrate's ability to meet its nutritional requirements and thereby survive, even in the absence of any chemical toxicant. Arrate, Rodriguez and Martinez-Madrid [96] investigated the relationship between amount of food supplements and water/sediment quality during a 28-day chronic and sub-chronic toxicity test, and found a direct correlation between food added, ammonia levels and invertebrate behavior and growth.

5.2.3. Factor: Is One Universal Formulation a Reasonable Objective?

Two studies have addressed the question whether it is reasonable to expect that a single synthetic sediment formulation is sufficiently versatile to meet all possible sediment test requirements. Fleming, Holmes and Nixon [68] and Gratzer and Ahlf [81] observed marked differences in sediment performance (i.e., binding of contaminant or bioavailability to macro-invertebrate test species) in response to varying the organic matter content of several synthetic sediment formulations. The latter group showed that native sediment could be better simulated by adjusting formulation components. Both groups concluded that it was unreasonable to expect one formulation to apply to all study objectives.

5.2.4. Factor: Acid-volatile sulfate (AVS) and Oxidation-Reduction Potential (ORP)

Gonzalez [57] incorporated synthetically-prepared metal sulfide salts into synthetic sediment formulations, and demonstrated that (depending on the cation used) synthetic AVS/sediment combinations behave quite similarly to their natural counterpart. The results of that study suggest that bulk synthetic sediment's ORP profile can be controlled consistently and systematically. The ability to control and systematically manipulate sediment ORP is another aspect of raising the level of realism in synthetic sediment formulations.

5.2.5. Factor: Conditioning Protocol(s)

Developing a realistic surrogate for the complex and dynamic medium that is aquatic sediment may never be feasible without addressing the issue of controlling equilibrium conditions in synthetic sediment formulations. Two options for addressing this issue are: (1) use synthetic components with characteristics that reflect "aged" components in natural sediment (e.g., use organic matter that has the character of humified or stabilized natural organic matter) or (2) include a conditioning period in the formulation protocol(s) wherein the synthetic formulation is allowed to "age" under controlled conditions (e.g., during which time labile components in the synthetic mixture presumably can stabilize or equilibrate).

A reference to a conference abstract was found in the literature [87], the subject of which was synthetic sediment conditioning, equilibration and component quality, and their influence on benthic invertebrate toxicity. Results similar to those briefly described in that conference abstract are found in a paper published the following year by the same investigators [89]. In that paper, they describe studying temporal changes in synthetic and natural sediment by measuring various physicochemical and biological parameters as a function of time. When comparing their synthetic materials to natural sediment aged for the same period of time, they observed that physical parameters related to hydrolysis equilibrated in 10 to 15 days.

5.2.6. Factor: Active Microbiological Populations in Synthetic Formulations

One of the most advanced factors in synthetic sediment development is the development and control of microbiological conditions in a completely fabricated matrix. Rudimentary attempts at addressing this complex factor have been limited to providing conditions for the synthetic substrate to recruit natural microbial populations from natural sources. This has been attempted either by (1) including natural organic matter (presumably containing indigenous microbiological populations) in the formulation, or (2) recruiting natural bacterial species into synthetic sediment by exposing it for a "conditioning period" to natural water containing indigenous microbial organisms. Data or results of the success of this method are limited.

Verrhiest et al. [79] monitored the microbiological activity in several samples of synthetic and natural sediment in response to toxicant concentrations and to various types and concentrations of organic matter. They reported that after allowing the synthetic sediment material to be inoculated with naturally recruited micro-organisms, the material successfully sustained some level of microbiological activity over the 4-week study period. Another group [108] published a study in which they compared microbiological community abundance and activity in synthetic sediment formulations versus those in natural sediment. Not surprisingly, the activity in synthetic sediment materials was less than that in natural sediment. They conclude that the microbiological factor could have direct influence on the effectiveness or usefulness of synthetic sediment for toxicological testing and study.

5.3. Novel Uses for Synthetic Sediment

Two studies found in the literature represent isolated examples of novel potential uses for synthetic sediment. One paper [88] reported the use of synthetic substrates for more accurately determining the fate (distribution) of cadmium within the system by using a stable isotope of the metal as a radio-isotope tracer. Using synthetic material in this study gave the investigators a *priori* and complete knowledge of available compartments and phases for contaminant distribution and partitioning. Another paper [109] used a synthetic sediment formulation as a well-characterized medium with which to develop an improved pore water extraction method.

6. DISCUSSION

This review confirms that the current status of synthetic sediment development is lethargic and deficient in precision and realism. After more than 45 years of effort, the collective energy for developing realistic synthetic sediment material has waned. Many presume that further progress in 3S development is intractable, and that the current state of 3S technology must be endured.

The progression of sediment assessment and SQM activity since the late 1980s (Table 1) implies an ever-expanding demand for greater measurement precision, statistical power and control of "background noise" in various types of sediment assessments. Synthetic sediment development has not kept pace with this demand. Because resources are often unavailable or diverted to other pressing needs, the sediment assessment community defaults to existing synthetic sediment formulations that are convenient but limiting in the value of data and information they can support.

This review seems to hint at a presumption unconsciously operating in the sediment assessment community: that the ultimate objective is to develop a single universal formulation that will satisfy any and all sediment assessment data quality objectives (DQO). The most common formulations currently in use were identified and reviewed. The components of those formulations, listed in Table 3, are ones used to create synthetic soil [133]. Those four- to six-components can not accurately simulate the magnitude and complexity of natural sediment CCF. Few investigators have attempted new materials or methods [57,75,89]. If this apparent presumption is real, such an expectation is a serious impediment to progress.

In light of the constraints on resources and impracticable expectations for synthetic sediment, perhaps future development can proceed by a “multiple-parallel” approach (i.e., multiple “standard” formulations, developed independent of each other, each developed for more narrowly defined purposes or functions). The universe of possible sediment assessment activities includes: culturing macroinvertebrates; investigating chemical-sediment interactions; testing sediment for chemical toxicity using sediment bioassays; studying physical habitat parameters for benthic organisms; identifying toxicity sources using sediment toxicity identification evaluations (TIEs) [12]. Each has activity-specific DQOs, and would likely require activity-specific procedures and tools. By extension, each of these activities might benefit from their own synthetic sediment, specifically tailored to meet their DQO-specific criteria. Each formulation can be standardized for its specific purpose, and its development would not be constrained by having to attain criteria related to other sediment assessments.

As an example, synthetic sediment used for culturing benthic fauna (a relatively less time-critical and complex activity) may not require a complex sediment formulation, or it may require organism-specific CCF that are not necessary for other sediment assessment activities. Further along the complexity continuum, sediment remediation decisions that rely on understanding the stressor identity, levels and effects within the unique context of local site conditions may call for a sophisticated sediment bioassay program to obtain the necessary information. Such scenarios would require sophisticated site-specific synthetic sediment formulations. Similarly, detailed study of site-specific nuances in physical-chemical sediment properties (e.g., [150, 151]) would benefit from highly sophisticated sediment materials.

As an experimental control treatment, such material could reduce background variability “noise” to sufficient extent that fine details in causal or correlative relationships can be discerned. As an experimental tool, investigators could accurately replicate observed conditions in field-collected sediment (e.g., simulating a contaminated field sediment by spiking a detailed synthetic sediment formulation), thereby confirming or refuting hypotheses with greater statistical power. These potential applications of synthetic sediment have been recognized for a long time [41, 42, 50], but the concept of developing multiple formulations, each standardized for a specific application, should be considered as a practicable path to attaining those long-envisioned goals.

This review also confirms the long-recognized importance of particulate organic matter CCF to synthetic sediment realism and utility, as it is to natural sediment [152]. Current practice of using bulk total organic carbon (TOC) concentrations as a normalizing parameter has performed the valuable service of allowing sediment scientists to model the influence of organic matter in natural sediment at a gross- or bulk-level [153, 154]. Used as an analytically pragmatic surrogate for the entire sediment organic matter phase (i.e., determined using laboratory furnaces or carbon analyzer instrumentation), TOC as a normalizing parameter is too coarse for deducing finer details in sediment processes. This is implied by the residual scatter in TOC-normalized data (e.g., [155]).

The inability to correlate bulk TOC concentrations to concentrations of true organic matter components in natural sediment, or to use TOC and total inorganic carbon data to deduce organic matter type is directly relevant to synthetic sediment development. Pulverized tree bark will interact with a contaminant very differently than will fish food flakes or commercial humic substances or charcoal. With advances in instrumentation and methods, we should see more examples appearing in the literature of sediment data modeling and analysis using actual organic components rather than the old TOC surrogate (i.e., lipids; [156-158]). In the context of synthetic sediment development, quantifying and qualifying sediment organic matter in new formulations can be achieved with methods other than bulk TOC. Working with specific organic components comparable to those found in natural sediment will significantly elevate the level of realism in synthetic sediment.

7. CONCLUSIONS

The 3S literature was reviewed to understand previous approaches and rationale, to instigate renewed interest in synthetic sediment development, and to provide suggestions for future progress. The results of this review provide the following information:

- The terminology in the 3S literature is imprecise and could be standardized to facilitate communication. Standard definitions are proposed.
- Use of surrogate materials in environmental studies is not new. They have been used in sediment assessments for over 45 years.
- The literature indicates that work in developing or

using 3S materials was sparse before 1990. The sudden and spontaneous increase in activity in the early 1990s has waned somewhat, likely due to lack of significant advances in synthetic sediment development.

- The basis for synthetic sediment development in the early 1990s likely was the existence of a synthetic soil matrix developed for soil toxicity testing using earthworms.
- 3S materials have been used in over 130 studies. They classify into one of four categories of study: physical sediment mechanics; sediment ecology (habitat); sediment toxicity; and sediment “bio-assimilation” (mostly bioaccumulation studies).
- The materials presumed to represent the silt component in almost all synthetic sediment formulations published to date were actually clay minerals. Thus, those formulations may have lacked a true silt component composed of non-clay minerals (e.g., quartz, oxides, carbonates, feldspar and mica, among others). The misrepresentation of silt by “large clay particles,” might have lead to misinterpretation of study results, particularly when study outcomes were correlated to mineral content.
- The organic matter content of synthetic sediment continues to be a major challenge to further progress in synthetic sediment development. Attempts to address this factor have been random and ineffective. What makes this factor so challenging is the simultaneous need for source/quality consistency and realistic complexity and heterogeneity.
- Factors such as inherent nutritional value of synthetic sediment, acid-volatile sulfide content, control of oxidation-reduction potential in synthetic sediment, reproducible conditioning and equilibration, and microbiological components represent potential areas for future development as the level of realism grows.
- There may be an underlying presumption to development work up to the present, namely that we need to identify or create one single universal material to meet any and all sediment assessment DQOs. This constraint may be hindering further progress.
- An alternative approach may be to develop assessment-specific or DQO-specific synthetic materials with controllable characteristics pertinent only to its intended use. This could open avenues for incorporating new materials into the synthetic sediment development process.

It is hoped that the information compiled in this review will challenge the status quo and spark renewed enthusiasm for developing synthetic sediment.

8. REFERENCES

[1] Adams WJ, Kimerle RA, Barnett JW. Sediment quality and aquatic life assessment. *Environ. Sci. Technol.*, 1992, 26: 1864-1875.

[2] Burton, GA, Jr. Assessing the toxicity of freshwater sediments. *Environ. Toxicol. Chem.*, 1991, 10: 1585-1627.

[3] Simpson SL, Batley GE, Chariton AA, Stauber JL, King CK, Chapman JC, Hyne RV, Gale SA, Roach AC, Maher WA. *Handbook for Sediment Quality Assessment*. Bangor, Australia: Centre for Environmental Contaminants Research (CSIRO), 2005.

[4] Canadian Council of Ministers of the Environment (CCME). *Protocol for the Derivation of Canadian Sediment Quality Guidelines for the Protection of Aquatic Life*. Ottawa: Environment Canada, 1995.

[5] Salmons W, Brils J. *Contaminated Sediments in European River Basins*. Eindhoven: European Sediment Research Network (SedNet), 2004. Found online at: http://www.sednet.org/download/Sednet_booklet_final.pdf.

[6] European Community. *Directive 2000/60/EC: the European Parliament and of the Council of 23 October 2000 establishing a framework for Community action in the field of water policy*. Official Journal of the European Communities, 2000, L.327/1. Found online at: http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2000:327:0001:0072:EN:PDF

[7] U.S. Environmental Protection Agency (USEPA). *An Overview of Sediment Quality in the United States*. Washington, D.C.: Office of Water, 1987.

[8] U.S. Environmental Protection Agency (USEPA). *The Incidence and Severity of Sediment Contamination in the Surface Waters of the United States: National Sediment Quality Survey* (2nd ed.). Washington, D.C.: Office of Science and Technology, 2004.

[9] U.S. Environmental Protection Agency (US EPA). *A Framework for Assessing and Reporting on Ecological Condition*. Washington, D.C.: Science Advisory Board, 2002.

[10] Hoffman, DJ, Rattner BA, Burton GA, Cairns J (eds.). *Handbook of Ecotoxicology* (2nd ed.). Boca Raton, Florida: Lewis Publishers, 2003.

[11] U.S. Environmental Protection Agency (USEPA). *Methods for Assessing the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Amphipods*. Washington, D.C.: Office of Research and Development, 1994.

[12] U.S. Environmental Protection Agency (USEPA). *Handbook: A Compendium of Chemical, Physical and Biological Methods for Assessing and Monitoring the Remediation of Contaminated Sediment Sites*. Duxbury, Massachusetts: Battelle Memorial Institute, 2003.

[13] Sokal R, Rohlf J. *Biometry: The Principles and Practices of Statistics in Biological Research* (3rd ed.). New York: W. H. Freeman, 1994.

[14] U.S. Environmental Protection Agency (USEPA). *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates*. Washington, D.C.: Office of Research and Development and Office of Water, 2000.

[15] American Standards for Testing and Materials (ASTM). *Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates*. West Conshohocken, Pennsylvania: ASTM International, 2010.

[16] Irving E, Major A. Post-depositional detrital remanent magnetization in a synthetic sediment. *Sedimentology*, 1964, 3: 135-143.

[17] Shirley DJ, Hampton LD. Shear-wave measurements in laboratory sediments. *J. Acoust. Soc. Amer.*, 1978, 63: 607-613.

[18] Barko JW, Smart RM. The nutritional ecology of *Cyperus esculentus*, an emergent aquatic plant, grown on different sediments. *Aquat. Bot.*, 1979, 6: 13-28.

[19] Smart RM, Barko JW. Nitrogen nutrition and salinity tolerance of *Distichlis spicata* and *Spartina alterniflora*. *Ecology*, 1980, 61: 630-638.

[20] Titus JA, Pfister RM. Effects of pH, temperature, and Eh on the uptake of cadmium by bacteria and an artificial sediment. *Bull. Environ. Contam. Toxicol.*, 1982, 28: 697-704.

[21] Barko JW, Smart RM. Effects of organic matter additions to sediment on the growth of aquatic plants. *J. Ecol.*, 1983, 71: 161-175.

[22] Beveridge TJ, Meloche JD, Fyfe WS. Diagenesis of metals chemically complexed to bacteria: Laboratory formation of metal phosphates, sulfides, and organic condensates in artificial sediment. *Appl. Environ. Microb.*, 1983, 45: 1094-1108.

[23] Mum DCG, Townsend BE, Lockhart WL. Bioavailability of six organic chemicals to Chironomus tentans larvae in sediment and water. *Environ. Toxicol. Chem.*, 1993, 2: 269-281.

[24] Taghon GL, Jumars PA. Variable ingestion rate and its role in optimal foraging behavior of marine deposit feeders. *Ecology*, 1984, 65: 549-558.

[25] Muir DCG, Rawn GP, Townsend BE, Lockhart WL, Greenhalgh R. Bioconcentration of cypermethrin, deltamethrin, fenvalerate and permethrin by *Chironomus tentans* larvae in sediment and water. *Environ. Toxicol. Chem.*, 1985, 4: 51-61.

[26] Kheboian C, Bauer CF. Accuracy of selective extraction procedures for metal speciation in model aquatic sediments. *Anal. Chem.*, 1987, 59: 1417-1423.

[27] Taghon GL. The benefits and costs of deposit feeding in the polychaete *Abarenicola pacifica*. *Limnol. Oceanogr.*, 1988, 33: 1166-1175.

[28] Arkin Y, Michaeli L. Strength and consistency of artificial clay – carbonate mixtures: Simulation of natural sediments. *Eng. Geol.*, 1989, 26: 201-213.

[29] Walsh GE, Weber DE, Brashers LK, Simon TL. Artificial sediments for use in tests with wetland plants. *Environ. Exp. Bot.*, 1990, 30: 391-396.

[30] Nix PG, Merry CH. Use of sediment bags as a monitor of fecal pollution in streams. *Bull. Environ. Contam. Toxicol.*, 1990, 45: 864-869.

[31] Hanes EC, Ciborowski JJH, Corkum LD. Standardized rearing materials and procedures for hexagenia, a benthic bioassay organism: comparison of sediment types. In: *Technology Transfer Conference on Environmental Research*. Toronto: Ontario Ministry of the Environment, 1990, p. 374-382.

[32] Walsh GE, Weber DE, Simon TL, Brashers LK. Toxicity tests of effluents with marsh plants in water and sediment. *Environ. Toxicol. Chem.*, 1991, 10: 517-525.

[33] Walsh GE, Weber DE, Esry LK, Nguyen MT. Synthetic sediments: a tool for research. In: *Proceedings of the Fifth Federal Interagency Sedimentation Conference*. Las Vegas: U.S.

Government Printing Office, 1991, 5-40 to 5-47.

[34] Ciborowski JJH, Hanes EC, Corkum LD. Standardized materials and procedures for Hexagenia, a benthic bioassay organism: Application to 21-day bioassays. In: *Technology Transfer Conference on Environmental Research*. Toronto: Ontario Ministry of the Environment, 1991, 676-684.

[39] Ciborowski JJH, Hanes EC, Corkum LD. Standardized rearing materials and procedures for Hexagenia, a benthic aquatic bioassay organism: serial dilution bioassay for sediment toxicity testing and organism calibration testing. In: *Technology Transfer Conference on Environmental Research*. Toronto: Ontario Ministry of the Environment, 1992; p.1-5.

[35] Walsh GE, Weber DE, Simon TL, Brashers LK, Moore JC. Use of marsh plants for toxicity testing of water and sediment. In: Gorsuch JW, Lower WR, Wang W, Lewis MA eds. *Plants for Toxicity Assessment: Second Volume*. Philadelphia: American Society for Testing and Materials, 1991, p. 341-354.

[36] Walsh GE, Weber DE, Nguyen MT, Esry LK. Responses of wetland plants to effluents in water and sediment. *Environ. Exp. Bot.*, 1991, 31: 351-358.

[37] Walsh GE, Weber DE, Esry LK, Nguyen MT, Noles J, Albrecht B. Synthetic substrata for propogation and testing of soil and sediment organisms. *Pedobiologia*, 1992, 36:1-10.

[38] Ciborowski JJH, Hanes EC, Corkum LD. *Standardized Rearing Materials and Procedures for Hexagenia, a Benthic Aquatic Bioassay Organism*. *Semi-Annual Progress Report to the Research Advisory Committee (Grant No. 450G-1992).* Toronto: Ontario Ministry of the Environment, 1992.

[39] Ciborowski JJH, Hanes EC, Corkum LD. Standardized rearing materials and procedures for Hexagenia, a benthic aquatic bioassay organism: serial dilution bioassay for sediment toxicity testing and organism calibration testing. In: *Technology Transfer Conference on Environmental Research*. Toronto: Ontario Ministry of the Environment, 1992; p.1-5.

[40] Burgess RM, Rogers BA, Rego SA, Corbin JM, Morrison GE. Sand spiked with copper as a reference toxicant material for sediment toxicity testing: A preliminary evaluation. *Arch. Environ. Contam. Toxicol.*, 1994, 26: 163-168.

[41] Suedel BC, Rodgers JH Jr. Development of formulated reference sediments for freshwater and estuarine sediment testing. *Environ. Toxicol. Chem.*, 1994, 13: 1163-1175.

[42] Suedel BC, Rodgers JH Jr. Responses of Hyalella azteca and Chironomus tentans to particle-size distribution and organic matter content of formulated and natural freshwater sediments. *Environ. Toxicol. Chem.*, 1994, 13: 1639-1648.

[43] Watzin MC, Roscigno PF, Burke WD. Community-level field method for testing the toxicity of contaminated sediments in estuaries. *Environ. Toxicol. Chem.*, 1994, 13: 1187-1193.

[44] Wynn, JC. *Artificial Sediment Formulation: A Combination of Materials to Mimic Natural Sediments*: *Research Report*. Oak Ridge: Department of Energy (DOE) Science and Engineering Research Semester, 1994.

[45] Frugis M. *Bioavailability of Metal Mixtures in Natural and Synthetic Sediments* (Master's Thesis). Boulder, Colorado: Colorado State University, 1995.

[46] Barrett KL. A comparison of the fate and effects of prochloraz in artificial and natural sediments. *J. Aquat. Ecosyst. Health*, 1995, 4: 239-248.

[47] Weber DE, Walsh GE, MacGregor MA. Use of vascular aquatic plants in phytotoxicity studies with sediments. In: Hughes JS, Biddinger GR, Mones E (eds.) *Environmental Toxicology and Risk Assessment* (3rd ed). ASTM STP 1218. Philadelphia: American Society for Testing and Materials, 1995, 187-200.

[48] Naylor C, Rodrigues C. Development of a test method for *Chironomus riparius* using a formulated sediment. *Chemosphere*, 1995, 31: 3291-3303.

[49] Leyes GA, Rose CA, Zhu W, Forster A, Bliss M. *Effects of Chlorothalonil on Chironomids and Dissipation in Laboratory Sediment-Water Systems*. In: Conference Abstract (unknown conference), 1996, pp 1-3.

[50] Suedel BC, Deaver E, Rodgers JH Jr.. Formulated sediment as a reference and dilution sediment in definitive toxicity tests. *Arch Environ. Contam. Toxicol.,* 1996, 30: 47-52.

[51] Bridges TS. Effects of organic additions to sediment, and maternal age and size, on patterns of offspring investment and performance in two opportunistic deposit-feeding polychaetes. *Mar. Biol.*, 1996, 125: 345-357.

[52] Lake JL, McKinney R, Osterman FA, Lake CA. C18-coated silica particles as a surrogate for benthic uptake of hydrophobic compounds from bedded sediment. *Environ. Toxicol. Chem.*, 1996, 15: 2284-2289.

[53] Suedel BC, Deaver E, Rodgers JH Jr. Experimental factors that may affect toxicity of aqueous and sediment-bound copper to freshwater organisms. *Arch. Environ. Contam. Toxicol.*, 1996, 30: 40-47.

[54] Weber DE, McKenney CL Jr, MacGregor MA, Celestial DM. Use of artificial sediments in a comparative toxicity study with larvae and post-larvae of the grass shrimp, Palaemonetes pugio. *Environ. Pollut.*, 1996, 93:129-133.

[55] Tiefenthaler LL, Skracic MA. Development of sediment bioassays using newly settled California halibut, *Paralichthys californicus*. Westminster, California: Section in an annual report, 1996.

[56] Hunter MA, Kan AT, Tomson MB. Development of a surrogate sediment to study the mechanisms responsible for adsorption / desorption hysteresis. *Environ. Sci. Technol.*, 1996, 30: 2278-2285.

[57] Gonzalez AM. A laboratory-formulated sediment incorporating synthetic acid-volatile sulfide. *Environ. Toxicol. Chem.*, 1996, 15: 2209-2220.

[58] Pringault O, de Wit R, Caumette P. A benthic gradient chamber for culturing phototrophic sulfur bacteria on reconstituted sediments. *FEMS Microbiol. Ecol.*, 1996, 20: 237-250.

[59] Harrahy EA, Clements WH. Toxicity and bioaccumulation of a mixture of heavy metals in Chironomus tentans (Diptera: Chironomidae) in synthetic sediment. *Environ. Toxicol. Chem.*, 1997, 16: 317-327.

[60] Gatenby, CM, Parker BC, Neves RJ. Growth and survival of juvenile rainbow mussels, Villosa iris (Lea, 1829) (Bivalva: Unionidae), reared on algal diets and sediment. *Amer. Malacol. Bull.*, 1997, 14: 57-66.

[61] Davies NA, Taylor MG, Simkiss K. The influence of particle surface characteristics on pollutant metal uptake by cells. *Environ. Pollut.*, 1997, 96: 179-184.

[62] Egeler P. Bioaccumulation of lindane and hexachlorobenzene by tubificid sludgeworms (oligochaeta) under standardised laboratory conditions. *Chemosphere*, 1997, 35: 835-852.

[63] Kan AT, Fu G, Hunter MA, Tomson MG. Irreversible adsorption of naphthalene and tetrachlorobiphenyl to Lula and surrogate sediments. *Environ. Sci. Technol.*, 1997, 31: 2176-2185.

[64] Meller M, Egeler P, Römbke J, Schallnass HJ, Nagel R, Streit B. Short-term toxicity of Lindane, Hexachlorobenzene, and copper sulfate to tubificid sludgeworms (Oligochaeta) in artificial media. *Ecotoxicol. Environ. Safety*, 1998, 39: 10-20.

[65] Clément B, Cadier C. Development of a new laboratory freshwater/ sediment microcosm test. *Ecotoxicology*, 1998, 7: 279-290.

[66] Pringault O, Kühl M, de Wit R, Caumette P. Growth of green sulphur bacteria in experimental benthic oxygen, sulphide, pH and light gradients. *Microbiology*, 1998, 144: 1051-1061.

[67] Güss S. Oxygen uptake at the sediment-water interface simultaneously measured using a flux chamber method and microelectrodes: Must a diffusive boundary layer exist? *Estuarine, Coastal, and Shelf Science*, 1998, 46: 143-156.

[68] Fleming RJ, Holmes D, Nixon SJ. Toxicity of Permethrin to Chironomus riparius in artificial and natural sediments. *Environ. Toxicol. Chem.*, 1998, 17: 1332-1337.

[69] Pyrak-Nolte LJ, Mullenbach BL, Li X, Nolte DD, Grader AS. Detecting sub-wavelength layers and interfaces in synthetic sediments using seismic wave transmission. *Geophys. Res. Lett.*, 1999, 26: 127-130.

[70] Lacey R, Watzin MC, McIntosh AW. Sediment organic matter content as a confounding factor in toxicity tests with Chironomus tentans. *Environ. Toxicol. Chem.*, 1999, 18: 231-236.

[71] Kemble NE, Dwyer FJ, Ingersoll CG, Dawson TD, Norberg-King TJ. Tolerance of freshwater test organisms to formulated sediments for use as control materials in whole sediment toxicity tests. *Environ. Toxicol. Chem.*, 1999, 18: 222-230.

[72] Wastlund D. *The Role of Sediment Characteristics and Food Regimen in a Toxicity Test with Chironomus Riparius* (Master’s Thesis). Uppsala, Sweden: Swedish University of Agricultural Sciences, 1999.

[73] Pringault O, de Wit R, Kühl M. A micro-sensor study of the interaction between purple sulfur and green sulfur bacteria in experimental benthic gradients. *Microb. Ecol.*, 1999, 37: 173-184.

[74] Davies NA, Edwards PA, Lawrence MAM, Simkiss K, Taylor MG. Biocide testing using

particles with controlled surface properties (artificial sediments). *Environ. Toxicol. Chem.*, 1999, 18: 2337-2342.

[75] Riberio R, Kelly LA, Goncalves F, Burton GA Jr., Soares AMVM. New artificial sediment for Chironomus riparius toxicity testing. *Bull. Environ. Contam. Toxicol.*, 1999, 63: 691-697.

[76] Schlekat CE, Decho AW, Chandler GT. Bioavailability of particle-associated silver, cadmium, and zinc to the estuarine amphipod Leptocheirus plumulosus through dietary ingestion. *Limnol. Oceanogr.*, 2000, 45: 11-21.

[77] Martínez-Tabche L, Gómez-Oliván LM, Martinez MG, Castillo CR, Santiago AM. Toxicity of nickel in artificial sediment on acetylcholinesterase activity and hemoglobin concentration of the aquatic flea, Moina macrocopa. *J. Environ. Hydrol.*, 2000, 8: 1-10.

[78] Quantin C, Lartiges SB, Munier-Lamy C, Ghanbaja J, Yvon J, Berthelin J. Microbial solubilisation of Cd from apatite during aerobic cellulose biodegradation. *Agron. Sustain. Dev.*, 2000, 20: 557-565.

[79] Verrhiest GJ, Clément B, Merlin G. Influence of sediment organic matter and fluoranthene-spiked sediments on some bacterial parameters in laboratory freshwater/ formulated sediment microcosms. *Aquat. Ecosyst. Health*, 2000, 3: 359-368.

[80] Lawrence MAM, Davies NA, Edwards PA, Taylor MG, Simkiss K. Can adsorption isotherms predict sediment bioavailability? *Chemosphere*, 2000, 41: 1091-1100.

[81] Gratzer H, Ahlf W. Adjustment of a formulated sediment for sediment testing with *Caenorhabditis elegans* (Nematoda). *Acta Hydroch. Hydrob.*, 2001, 29: 41-46.

[82] McDonald BG, Haynes PA. Silica sand as an artificial control sediment in a 20 day *Neanthes arenaceodentata* toxicity test. *Environ. Toxicol.* 2001, 16: 172-176.

[83] Verrhiest GJ, Clément B, Blake G. Single and combined effects of sediment-associated PAHs on three species of freshwater macro-invertebrates. *Ecotoxicology*, 2001, 10: 363-372.

[84] Hagopian-Schlekat TG, Chandler T, Shaw TJ. Acute toxicity of five sediment-associated metals, individually and in a mixture, to the estuarine meiobenthic harpacticoid copepod *Amphiascus tenuiremis*. *Mar. Environ. Res.*, 2001, 51: 247-264.

[85] Simkiss K, Davies NA, Edwards PA, Lawrence MAM, Taylor MG. The use of sediment analogues to study the uptake of pollutants by chironomid larvae. *Environ. Pollut.*, 2001, 115: 89-96.

[86] Fan W, Wang WX. Sediment geochemical controls on Cd, Cr, and Zn assimilation by the clam *Ruditapes philippinarum*. *Environ. Toxicol. Chem*, 2001, 20: 2309-2317.

[87] Verrhiest GJ, Clément B, Lamy-Enrici MH, Montuelle B, Garric J. Influence of sediment type on the toxicity of polycyclic aromatic hydrocarbons on aquatic invertebrates. In: *International Conference*. Amsterdam: Aquat Ecosyst Health Society, 2001, p 1.

[88] Eimers MC, Evans RD, Welbourn PM. Partitioning and bioaccumulation of cadmium in artificial sediment systems: application of a stable isotope tracer technique. *Chemosphere*, 2002, 46: 543-551.

[89] Verrhiest GJ, Cortes S, Clément B, Montuelle B. Chemical and bacterial changes during laboratory conditioning of formulated and natural sediments. *Chemosphere*, 2002, 46: 961-974.

[90] Lu Y, Riyanto N, Weavers LK. Sonolysis of synthetic sediment particles: Particle characteristics affecting particle dissolution and size reduction. *Ultrason. Sonochem.*, 2002, 9: 181-188.

[91] Williamson KS, Petty JD, Huckins JN, Lebo JA, Kaiser EM. Sequestration of priority pollutant PAHs from sediment pore water employing semi-permeable membrane devices. *Chemosphere*, 2002, 49: 717-729.

[92] Péry ARR, Mons R, Flammarion P, Lagadic L, Garric J. A modeling approach to link food availability, growth, emergence, and reproduction for the midge *Chironomus riparius*. *Environ. Toxicol. Chem*,. 2002, 21: 2507-2513.

[93] Goedkoop W, Peterson M. The fate, distribution, and toxicity of lindane in tests with *Chironomus riparius*: Effects of bioturbation and sediment organic matter content. *Environ. Toxicol. Chem.*, 2003, 22: 67-76.

[94] Besser JM, Brumbaugh WG, May TW, Ingersoll CG. Effects of organic amendments on the toxicity and bioavailability of cadmium and copper in spiked formulated sediments. *Environ. Toxicol. Chem.*, 2003, 22: 805-815.

[95] Pery ARR, Sulmon V, Mons R, Flammarion P, Lagadic L, Garric J. A model to understand the confounding effects of natural sediments in

toxicity tests with *Chironomus riparius*. *Environ. Toxicol. Chem.*, 2003, 22: 2476-2481.

[96] Arrate JA, Rodriguez P, Martinez-Madrid M. Tubifex tubifex chronic toxicity test using artificial sediment: Methodological issues. *Limnetica*, 2004, 23: 25-36.

[97] Organization for Economic Cooperation and Development (OECD). *Guidelines for the Testing of Chemicals, Section 2: Effects on Biotic Systems – Test No. 218: Sediment-Water Chironomid Toxicity Using Spiked Sediment*. Paris: OECD Publishing, 2004.

[98] Trulleyová S, Rulík M. Determination of biodegradable dissolved organic carbon in waters: comparison of batch methods. *Sci. Total. Environ.*, 2004, 332: 253-260.

[99] Clément B, Devaux A, Perrodin Y, Danjean M, Ghidini-Fatus M. Assessment of sediment ecotoxicity and genotoxicity in freshwater laboratory microcosms. *Ecotoxicology*, 2004, 13: 323-333.

[100] Olvera-Hernández E, Martinez-Tabche L, Martinez-Jerómino F. Bioavailability and effects of Malathion in artificial sediments on *Simocephalus vetulus* (Cladocera: Daphniidae). *Bull. Environ. Contam. Toxiol.*, 2004, 73:197-204.

[101] Hruska KA, Dubé MG. Using artificial streams to assess the effects of metal-mining effluent on the life cycle of the freshwater midge (*Chironomus tentans*) in-situ. *Environ. Toxicol. Chem.*, 2004, 23: 2709-2718.

[102] McComb P, Black K. Detailed observations of littoral transport using artificial sediment tracer, in a high-energy, rocky reef and iron sand environment. *J. Coastal Res.*, 2005, 21: 358-373.

[103] Liebig M, Egeler P, Oehlmann J, Knacker T. Bioaccumulation of 14C-17a-ethinylestradiol by the aquatic oligochaete *Lumbriculus variegatus* in spiked artificial sediment. *Chemosphere*, 2005, 59: 271-280.

[104] Kim SD, Bae JE, Park HS, Cha DK. Bioleaching of cadmium and nickel from synthetic sediments by *Acidithiobacillus ferrooxidans. Environ. Geochem. Hlth.*, 2005, 27: 229-235.

[105] Bishop MJ. Artificial Sampling Units: A tool for increasing the sensitivity of tests for impact in soft sediments. *Environ. Monit. Assess.*, 2005, 107: 203-220.

[106] Hruska KA, Dubé MG. Comparison of a partial life-cycle bioassay in artificial streams to a standard beaker bioassay to assess effects of metal mine effluent on *Chironomus tentans*. *Environ. Toxicol. Chem*,. 2005, 24: 2325-2335.

[107] Miño LA, Folco S, Pechén de D'Angelo AM, Verrengia Guerrero NR. Modeling lead bioavailability and bioaccumulation by *Lumbriculus variegatus* using artificial particles. Potential use in chemical remediation processes. *Chemosphere*, 2005, 63: 261-268.

[108] Goedkoop W, Widenfalk A, Haglund AL, Steger K, Bertilsson S. Microbial characterization of artificial sediment and comparisons with natural sediments: implications for toxicity testing. *Environ. Toxicol. Chem.*, 2005, 24: 2725-2733.

[109] Lopes I, Riberio R. Optimization of a pressurization methodology for extracting pore-water. *Chemosphere*, 2005, 61: 1505-1511.

[110] Doig LE, Liber K. Nickel partitioning in formulated and natural freshwater sediments. *Chemosphere*, 2006, 62: 968-979.

[111] Liu Z, Lee CM. Drying effects on sorption capacity of coastal sediment: The importance of architecture and polarity of organic matter. *Geochim. Cosmochim. Acta*, 2006, 70: 3313-3324.

[112] Amaya-Chávez A, Martinez-Tabche L, López-López E, Galar-Martínez M. Methyl parathion toxicity to and removal efficiency by *Typha latifolia* in water and artificial sediment. *Chemosphere*, 2006, 63: 1124-1129.

[113] Gaskell, PN, Brooks AC, Maltby L. Variation in the bioaccumulation of a sediment-sorbed hydrophobic compound by benthic macro-invertebrates: Patterns and mechanisms. *Environ. Sci. Technol.*, 2007, 41: 1783-1789.

[114] Roman YE, De Schamphelaere KAC, Nguyen LTH, Janssen CR. Chronic toxicity of copper to five benthic invertebrates in laboratory-formulated sediment: Sensitivity comparison and preliminary risk assessment. *Sci. Total. Environ.*, 2007, 387: 128-140.

[115] Organization for Economic Cooperation and Development (OECD). *Guidelines for the Testing of Chemicals, Section 2: Effects on Biotic Systems – Test No. 225: Sediment-water Lumbriculus Toxicity Test using Spiked Sediment*. Paris: OECD Publishing, 2007.

[116] Zhong H, Wang WX. Methylmercury extraction from artificial sediments by the gut juice of the sipunculan, Sipunculus nudus. *Environ. Toxicol. Chem.*, 2008, 27: 138-145.

[117] Mauclair C, Layshock J, Carpi A. Quantifying

the effect of humic matter on the emission of mercury from artificial soil surfaces. *Appl. Geochem.*, 2008, 23: 594-601.

[118] Beg KR, Ali S. Toxicity of reference sediment samples. *Amer. J. Environ. Sci.*, 2008, 4: 347-352.

[119] Sachs S, Bernhard G. Sorption of U(VI) onto an artificial humic substance-kaolinite association. *Chemosphere*, 2008, 72: 1441-1447.

[120] Cantwell MG, Burgess RM, King JW. Resuspension of contaminated field and formulated reference sediments. Part I: Evaluation of metal release under controlled laboratory conditions. *Chemosphere*, 2008, 73: 1824-1831.

[121] Organization for Economic Cooperation and Development (OECD). *Guidelines for the Testing of Chemicals, Section 3: Degradation and Accumulation – Test No. 315: Bioaccumulation in sediment-dwelling benthic oligochaetes.* Paris: OECD Publishing, 2008.

[122] Karlsson J, Sundberg H, Åkerman G, Grunder K, Eklund B, Breitholtz M. Hazard identification of contaminated sites: ranking potential toxicity of organic sediment extracts in crustacean and fish. *J. Soils Sediments*, 2008, 8: 263-274.

[123] Oviedo-Gómez DGC, Galar-Martínez M, García-Medina S, Razo-Estrada C, Gómez-Oliván LM. Diclofenac-enriched artificial sediment induces oxidative stress in *Hyalella azteca*. *Environ. Toxicol. Pharm.*, 2009, 29: 39-43.

[124] Brooks AC, Gaskell PN, Maltby LL. Importance of prey and predator feeding behaviors for trophic transfer and secondary poisoning. *Environ. Sci. Technol.*, 2009, 43: 7916-7923.

[125] Marinkovic M, Kraak MHS. Development of an easily made artificial sediment that reduces experimental variability. In: *Proceedings of the Netherlands Entomological Society Meeting*. Amsterdam: Nederlandse Entomologische Vereniging, 2009, 95-101.

[126] Åkerblom N, Goedkoop W, Nilsson T, Kylin H. Particle-specific sorption desorption properties determine test compound fate and bioavailability in toxicity tests with *Chironomus riparius*: High-resolution studies with lindane. *Environ. Toxicol. Chem.*, 2010, 29: 1520-1528.

[127] Slootweg T, Alvinerie M, Egeler P, Gilberg D, Kukkonen JVK, Oehlmann J, Prasse C, Sormunen A, Liebig M. Bioaccumulation of ivermectin from natural and artificial sediments in the benthic organism *Lumbriculus variegatus*. *J. Soils Sediments*, 2010, 10: 1611-1622.

[128] Organization for Economic Cooperation and Development (OECD). *Guidelines for the Testing of Chemicals, Section 3: Degradation and Accumulation – Test No. 233: Sediment-Water Chironomid Life-Cycle Toxicity Test Using Spiked Water or Spiked Sediment*. Paris: OECD Publishing, 2010.

[129] Nasi M, Piol M, Di Risio C, Verrengia Guerrero NR. Investigating arsenic bioavailability and bioaccumulation by freshwater oligochaete *Lumbriculus variegatus*. *Arch. Environ. Contam. Toxicol.*, 2011, 61: 426-434.

[130] Gillett JW, Gile JD. Pesticide fate in terrestrial laboratory ecosystems. *Int. J. Environ. Stud.*, 1976, 10: 15-22.

[131] Gile JD, Gillett JW. Fate of selected fungicides in a terrestrial laboratory ecosystem. *J. Agric. Food Chem.*, 1979, 27: 1159-1164.

[132] Gile JD, Collins JC, Gillett JW. Fate of selected herbicides in a terrestrial laboratory microcosm. *Environ. Sci. Technol.*, 1980, 14: 1124-1128.

[133] Organization for Economic Cooperation and Development (OECD). *Guidelines for the Testing of Chemicals, Section 2: Effects on Biotic Systems – Test No. 207: Earthworm, Acute Toxicity Tests*. Paris: OECD Publishing, 1984.

[134] Organization for Economic Cooperation and Development (OECD). *Guidelines for the Testing of Chemicals, Section 2: Effects on Biotic Systems – Test No. 222: Earthworm Reproduction Test (Eisenia fetida).* Paris: OECD Publishing, 2004.

[135] Organization for Economic Cooperation and Development (OECD). *Guidelines for the Testing of Chemicals, Section 3: Degradation and Accumulation – Test No. 317: Bioaccumulation in Terrestrial Oligochaetes*. Paris: OECD Publishing, 2010.

[136] Van Gestel CAM, van Dis WA, van Breeman EM, Sparenburg PM. Development of a standardized reproduction toxicity test with the earthworm species *Eisenia fetida Andrei* using copper, pentachlorophenol and 2,4-dichloroaniline. *Ecotox. Environ. Safe.*, 1989, 18: 305-312.

[137] Crommentuijn T, Brils J, Vanstraalen NM. Influence of cadmium on life-history characteristics of *Folsomia candida* (Willem) in an artificial soil substrate. *Ecotox. Environ. Safe.*, 1993, 26: 216-227.

[138] Tucker B, McLean JE, Brar JS, Anderson AJ. Soil composition and suppression of pyrene mineralization by *Phanerochaete chrysosporium.* In: Hazardous Waste Research, *Proceedings of the 10th Annual Conference on Hazardous Waste Research*. Manhattan, Kansas: Kansas State University, 1996, Volume 1, 325-332.

[139] Ali NA, Ater M, Sunahara GI, Robidoux PY. Phytotoxicity and bioaccumulation of copper and chromium using barley (*Hordeum vulgare L.*) in spiked artificial and natural forest soils. *Ecotox. Environ. Safe.*, 2004, 57: 363-374.

[140] Adams MS, Stauber JL. Development of a whole-sediment toxicity test using a benthic marine microalga. *Environ. Toxicol. Chem.*, 2004, 23: 1957-1968.

[141] Haden WL Jr. *Attapulgite: Properties and Uses*. In: National Clay Committee, *Tenth National Conference on Clays and Clay Minerals*. Austin, Texas: National Academy of Sciences, 1963, Volume 1, 284-290. Found online at: http://www.clays.org/journal/archive/volume%2010/10-1-284.pdf

[142] BASF. *Attapulgite: Colloidal Hydrated Magnesium Alumino-Silicate*: BASF brochure. Florham Park, NJ: BASF Chemical Company, 2007. Found online at: http://www.supmat.com/download/BASF/Attapulgite%20Final%2011.15.2007.pdf

[143] BASF. *Safety Data Sheet: ASP®400* (Version 1.1). Florham Park, NJ: BASF Chemical Company, 2010. Found online at: http://www.hmroyal.com/msds/ASP-400.pdf

[144] Baker JE, Capel PD, Eisenreich SJ. Influence of colloids on sediment-water partition coefficients of polychlorobiphenyl congeners in natural waters. *Environ. Sci. Technol.*, 1986, 20: 1136-1143.

[145] Verkhovets IA, Chizhikova NP, Vladychenskii AS. Mineralogical composition of silt fractions and its transformation under the impacts of different cenoses in model lysimeters. *Eurasian Soil Sci.*, 2006, 39: 528-538.

[146] Winterwerp JC, Van Kesteren WGM. *Introduction to the Physics of Cohesive Sediment in the Marine Environment*. Amsterdam: Elsevier, 2004.

[147] Wang YH. The Intertidal erosion rate of cohesive sediment: A case study from Long Island Sound. *Estuar. Coast. Shelf. Sci.*, 2003, 56: 891-896.

[148] Allen JRL. *Principles of Physical Sedimentology*. London: Allen & Unwin, 1985.

[149] McCave IN, Manighetti B, Robinson SG. Sortable silt and fine sediment size/ composition slicing: Parameters for palaeocurrent speed and palaeoceanography. *Paleoceanography*, 1995, 10: 593-610.

[150] Rowan DJ. *The Distribution, Texture and Trace Element Concentrations of Lake Sediments* (Doctoral dissertation). Montreal, Canada: Department of Biology, McGill University, October 1991.

[151] Rowan DJ, Kalff J, Rasmussen JB. Estimating the mud deposition boundary depth in lakes from wave theory. *Can. J. Fish Aquat. Sci.*, 1992, 49: 2490-2497.

[152] Granberg ME, Selck H. Effects of sediment organic matter quality on bioaccumulation, degradation, and distribution of pyrene in two macrofaunal species and their surrounding sediment. *Mar. Environ. Res.*, 2007, 64: 313-335.

[153] Di Toro DM, Zarba C, Hansen DJ, Berry W, Swartz RC, Cowan CE, Pavlou SP, Allen HE. Technical basis for establishing sediment quality criteria for nonionic organic chemicals using equilibrium partitioning. *Environ. Toxicol. Chem.*, 1991, 10: 1541-1583.

[154] U.S. Environmental Protection Agency (USEPA). *Technical Basis for Deriving Sediment Quality Criteria for Nonionic Organic Contaminants for the Protection of Benthic Organisms by using Equilibrium Partitioning* (EPA 822-R-93-011). Washington, D.C.: Office of Water, 1993.

[155] Delbeke K, Claude RJ, Bossicart M. Organochlorides in different fractions of sediments and different planktonic compartments of the Belgian Continental Shelf and Scheldt Estuary. *Environ. Pollut.*, 1990, 66: 325-349.

[156] Brunson EL, Canfield TJ, Dwyer FJ, Ingersoll CG, Kemble NE. Assessing the bioaccumulation of contaminants from sediments of the Upper Mississippi River using field-collected oligochaetes and laboratory-exposed *Lumbriculus variegatus*. *Arch. Environ. Contam. Toxicol.*, 1998, 35: 191–201.

[157] Ingersoll CG, Brunson EL, Wang N, Dwyer FJ, Ankley GT, Mount DR, Huckins J, Petty J, Landrum PF. Uptake and depuration of nonionic organic contaminants from sediment by the oligochaete *Lumbriculus variegatus*. *Environ. Toxicol. Chem.*, 2003, 22: 872–885.

[158] Pickard SW, Clarke JU. Benthic bioaccumulation and bioavailability of polychlorinated dibenzo-p-dioxins/dibenzofurans from surficial Lake Ontario sediment. *J. Great Lakes Res.*, 2008, 34: 418–433.

AES 120619