DETERMINATION OF CLEANABILITY OF PLASTIC SURFACES

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ACADEMIC DISSERTATION
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ABSTRACT

The cleanability of plastic materials can be studied from the molecular scale to practical scale. The present study concentrated on poly(vinyl chloride) (PVC) model materials and commercial plastic materials by focusing on the following areas of cleanability: First, to evaluate the feasibility of a colorimetric method. Second, to establish a radiotracer technique to investigate soil accumulation and to evaluate the effect of the composition of PVC model materials on accumulation. Third, to examine the feasibility of a contact angle measurement technique to determine the surface properties of commercial plastic materials and then to evaluate these surface properties related to cleanability. Fourth, to evaluate the feasibility of foil sampling and optical methods to measure dust accumulation on various furniture and floor surfaces in office buildings.

The colorimetric measurement proved to be a reproducible method for evaluating the cleanability of plastic materials in this study design with various soils and surface materials. The results were utilized to evaluate the relationship between cleanability and the surface properties of plastic materials.

The non-destructive radiotracer technique with the gamma-ray emitter ^{51}Cr provided information on the amount of soil at the molecular level. The method was suitable for investigating soil accumulation into PVC model surfaces during successive soiling-cleaning cycles. The amount of the plasticizer of PVC model materials influenced soil accumulation.

The contact angle measurement technique appeared to be feasible. The contact angle predicted the cleanability of both particle- and oil-soiled plastic materials. In addition, the polar component of surface free energy appeared to be related to the cleanability of plastic materials. The most easily cleanable materials had the lowest contact angle and the highest polar component of surface free energy regardless of the soil type used.

In a study in office environments, cleanability of surface materials was determined using foil sampling followed by an optical method. The method could identify differences in dust accumulation between different types of surface, thus allowing the practical assessment of cleanability of surface materials for the evaluation of cleaning methods and programmes.

Keywords: plastic, contact angle measurement, radiochemical tracer technique, colorimetry, cleaning, soiling, wettability, surface free energy

FOREWORD

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Eija Pesonen-Leinonen

ABBREVIATIONS

ADSA-P	axisymmetric drop shape analysis - profile
ASTM	American Society for Testing and Materials
CIE	Commission Internationale de l'Eclairage (International Lighting Commission)
CIELAB	CIE L*a*b* color space
cp5m	counts per five minutes
DDB	plasticizer, dodecylbenszene
DEHP	plasticizer, di-2-ethylhexyl phthalates, DOP
DIHP	plasticizer, di-isoheptyl phthalate
DINP	plasticizer, di-isonyl phthalate
DOP	plasticizer, dioctyl phthalate, DEHP
DS	Dansk Standard
EN	European Standard
ELPI	Elinympäristön pintojen hallinta (Control of surfaces in everyday life) – project
ESD	electrostatic discharge
DINCH	plasticizer, Hexamoll DINCH, di-isononyl-cyclohexane-1,2-dicarboxylate
INSTA	Inter Nordic Standard
L*	lightness L* scale measured as blackness-whiteness
L*a*b*	color space, the scale is defined by a vertical axis and two intersecting perpendicular axes
LAS	surfactant, linear alkylbenzenesulfonate
MESERAN	Measurement and Evaluation of Surfaces by Evaporative Rate Analysis
PAH	polycyclic aromatic hydrocarbons
phr	parts per hundred parts of resin (e.g. PVC) by weight
PM	particulate matter
POP	polyolefin plastomers
PUR	polyurethane
PVC	vinyl, poly(vinyl chloride)
SBS	Sick Building Syndrome
SFE	surface free energy
UV	ultra violet
VOC	volatile organic compounds

wt%	weight percent
θ	contact angle
θ_A	advancing contact angle,
θ_R	receding contact angle
θ_S	static contact angle
γ_S	solid surface free energy

LIST OF ORIGINAL PUBLICATIONS

The thesis is based on the following articles, which are referred to in the text by their Roman numerals:

I. **Pesonen-Leinonen, E., Kuisma, R., Redsven, I., Sjöberg A.-M. & Hautala, M**. Can contact angle measurements be used for predicting soiling and cleaning tendency of plastic flooring materials? Contact Angle, Wettability and Adhesion. *Submitted.*

II. **Pesonen-Leinonen, E., Kuisma, R., Redsven, I., Sjöberg A.-M. & Hautala, M.** 2005. Cleanability of plastic flooring materials related to their surface properties. Tenside Surfactants Detergents 42 (3), 148-153.

III. **Pesonen-Leinonen, E., Redsven, I., Neuvonen, P., Hurme, K.-R., Pääkkö, M., Koponen, H.-K., Pakkanen, T.T., Uusi-Rauva, A., Hautala, M. & Sjöberg, A.-M.** 2005. Radioisotope technique to determine the cleanability of surfaces. Applied Radiation and Isotopes. *In Press*.

IV. **Pesonen-Leinonen, E., Redsven, I., Kuisma, R., Hautala, M. & Sjöberg, A.-M.** 2003. Cleaning efficiency of mop cloths on floor coverings. Tenside Surfactants Detergents 40 (2), 80-86.

V. **Pesonen-Leinonen, E., Tenitz, S. & Sjöberg, A.-M.** 2004. Surface Dust Contamination and Perceived Indoor Climate in Office Buildings. Indoor Air 14 (5), 317-324.

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1 INTRODUCTION

The cost of cleaning constitutes a considerable part of the total cost of building operations and maintenance. Cleaning costs are estimated to represent about one third of the operating costs of buildings. The demand for improved cleanability of surface materials is continuously topical. In Europe plastic materials are one of most used floor and wall materials in many public buildings (Powel et al. 2000). One solution to reduce soiling has been to develop soil repellent and self-cleaning coatings and materials. The choice of material for a given function requires knowledge of the cleanability properties of surface materials. The importance of selecting soil-resistant materials has been recognized. For example, the standard EN 14565 “Resilient floor coverings – Floor covering based upon synthetic thermoplastic polymers – Specification” was published in 2004. This emphasizes the need to characterize the soilability of flooring materials before procurement. In January 2002 Tekes (the National Technology Agency of Finland) started the technology programme Clean Surfaces 2002-2006 (PINTA in Finnish), the primary goal of which has been to create comprehensive understanding of the basic phenomena in the chemistry and physics of clean and dirty surfaces.

Soiling and cleaning are complex subject. Consequently, the determination of cleanability is a versatile process including surface characterisation, soiling and cleaning processes with numerous parameters and soil determination methods. Many determination methods of cleanliness exist for various purposes of use and requirements, ranging from quantifying molecular level methods in precision cleaning to qualitative visual evaluation methods in practical situations.

The present study is a part of the ELPI (Control of surfaces in everyday life) project in the Clean Surfaces programme of Tekes. The task of the ELPI –project groups is to acquire knowledge on soiling and cleaning phenomena, firstly by developing methods for soiling, cleaning and soil determination and secondly by modifying surface properties of plastic and ceramic materials to be more soil-resistant. In this context, the primary objectives of this study were to develop and implement the determination methods of cleanability, to acquire knowledge of the physicochemical surface properties of commercial plastic materials using contact angle measurements and the effect of composition of PVC model

materials on soil accumulation, and to evaluate foil sampling and optical methods to measure the amount of dust on surfaces in office environments. The effect of the topography of these plastic materials on soilability and cleanability is discussed by Kuisma et al. (2005a, b).

2 REVIEW OF THE LITERATURE

2.1 PLASTIC SURFACE MATERIALS

Plastics and other resilient materials are commonly used as floor and wall coverings in indoor environments. One of the most frequently used materials is poly(vinyl chloride) (PVC) flooring, also known as “vinyl” flooring (Brydson 1999). In the year 2000 the total consumption of PVC flooring and wall materials was about 6 million square meters, which was 83 % of the total consumption of resilient coverings (linoleum, plastics, corks and rubber) and wall surface materials and 34 % of all surface materials in Finland (including carpets, resilient coverings, timbers and ceramics) (Eiro 2001).

The main types of PVC floorings are vinyl tiles or sheets, and vinyl composition tiles. The surface of vinyl floorings can be smooth or embossed with or without a permanently bonded coating. Vinyl floorings are classified into homogeneous and heterogeneous vinyl floorings. Furthermore, vinyl sheet floorings are classified as to wear type, backing type, minimum wear layer thickness and minimum overall thickness (Levy 2001). In addition, plastic floorings include monolithic impermeable floors which are placed on a concrete slab. The most traditional floors are made of acrylic, epoxy, polyurethane, polyester or vinyl ester resins (Hendley 1995). Plastic flooring materials are a heterogeneous group of surface materials with many applications. They are available for varying uses in homes or in public buildings or for special rooms, e.g. sport halls. Examples of these applications are impact-sound insulating, electrostatic conductive, static dissipative, foam backed or enhanced slip retardant surfaces.

2.1.1 Components of plastic materials and cleanability

PVC plastic materials and their main components are the focus of this section. Other plastic materials are briefly considered. Vinyl floorings are made of poly(vinyl chloride), and various compounds are added to tailor the characteristics of the final product, such as colour, flexibility, hardness and sheen. They may contain the following ingredients: polymers, plasticizers, stabilisers, fillers, pigments, polymeric processing aids and impact modifiers (Brydson 1999). Vinyl flooring materials contain the following components: PVC as a binder (24-47 wt%), plasticizers (6-22 wt%), stabilisers (<2 wt%), fillers (30-70 wt%), pigments (1-2 wt%) and others (<2 wt%). For environmental reasons, PVC flooring materials have recently faced competition from other polymers such as polyolefins (Hloch et al. 2003).

Poly(vinyl chloride)

PVC as a thermoplastic consists of individual polymer chains. The polymer is colourless and rigid, with low elongation and some degree of brittleness which is typical for glassy polymers, and with limited heat stability (Ram 1997; Brydson 1999). All vinyl products are made from combinations of vinyl resin, poly(vinyl chloride) or a copolymer of vinyl chloride with other monomeric materials, and various additives that give these products their particular properties (Levy 2001).

Plasticizers

Plasticizers help processing of plastic materials and modify the final product (Sears and Darby 1982). They lower the melt viscosity and alter the physical properties of the product, causing increased softness, flexibility and elongation and decreased the cold flex temperature (Brydson 1999). Phthalate plasticizers are widely used in many PVC applications (Wadey 2003). One of most used phthalate plasticizers is di-2-ethylhexyl phthalate (DEHP or DOP, dioctyl phthalate) (Brydson 1999). Plasticizers have direct effects on the stain resistance of materials (Colletti et al. 1998). In addition, the structure, volatility, concentration, extraction resistance and solubility parameter of plasticizers affect staining (Colletti et al. 1998). Stain resistance of plasticizers to various oily and coloured soils depends on the stain and differs between vinyl flooring types (sheet and composite) (Bohnert et al. 1998). The best stain resistance among typical plasticizers is

imparted by materials with the best compatibly with PVC (Sears and Darby 1982). They remain a part of the material and do not migrate to the surface of the PVC wear layer (Colletti et al. 1998).

Recently, there has been increasing interest in alternative plasticizers, e.g. benzoates, that improve both processing and performance properties, such as stain resistance and UV light stability, and have no environmental or toxicological concerns (Bohnert et al. 1999). Another new alternative plasticizer is Hexamoll DINCH (di-ester-based cyclohexane-dicarboxylic acid) (Sparrow 2002) or soy oil derived plasticizers (Vijayendran et al. 2001). Hexamoll DINCH was developed especially for sensitive applications (Wadey 2003). Recently, the following plasticizers or their mixtures have been used in flooring materials: benzoate, DINP (di-isonyl phthalate), DIHP (di-isoheptyl phthalate) and DDB (dodecylbensen).

Stabilizers

Stabilizers are used to minimize degradation and discoloration due to heat and light, and to improve the resistance to weathering, particularly degradation by ultraviolet radiation (Brydson 1999). Barium-zinc-soaps and epoxised soybean oil are one of most used stabilizers in vinyl flooring materials.

Fillers

Fillers are commonly employed in order to reduce costs or for technical reason such as to increase the hardness of a flooring compound (Brydson 1999). Various calcium carbonates (e.g. limestone), unsaturated polyester (Ram 1997) and sand (Wypych 2000), e.g. quartz silica, have been used as fillers in vinyl composite tiles. The fillers also affect soiling tendency (Sears and Darby 1982), in rather different ways. Fillers with more hydrophilic surfaces tend to resist staining by oil-soluble stains but are more susceptible to water-soluble stains (Sears and Darby 1982; Brydson 1999).

Other additives

Pigments such as titanium oxides and other oxides are added during the manufacturing process to give vinyl a range of colours. Among the most useful pigments are titanium dioxide and various iron oxides (Ram 1997). Carbon black is added to plastic materials to modify their electrostatic properties, in order to reduce static build-up (Sears and Darby

1982). Electrostatic discharge (ESD) materials attract less dust particles to their surface than insulative materials (Lesniewski and Yates 2001).

Coatings and treatments of plastic materials

The cleanability of plastic materials is improved by different coatings or treatments. The clear coating or wear layer of low-plasticized PVC on vinyl flooring material improves cleanability at least against stains (Sears and Darby 1982). Floor-care polymers based on pure acrylics or styrene acrylics are often applied on PVC floor surfaces to facilitate maintenance. These polymers have e.g. good water resistance and resistance to block and heel marking, scuffing and dirt pick-up (Ifran 1998). In recent years, cleanability of PVC flooring material has been improved by the addition of a thin film of PUR (<1 wt%) on the top of the surface. The chemical composition of the PUR film varies and some compositions include acrylic. These are also polyurethane coatings, which may be formulated either as one-component or two-component coatings. They have good gloss, hardness, flexibility, abrasion resistance, chemical resistance and ultraviolet (UV) durability (Chen and Wojcik 2000) and are easily cleanable (Temtchenko et al. 2001).

If only the topmost layer of flooring material is considered, some linoleum floorings could be regarded as a plastic surface because of their acrylate layer on the surface of the material. However, it should be taken into account that the whole bulk composition can affect soilability and cleanability and that the acrylic layer may become worn.

Polyolefins

Poly(vinyl chloride) has recently experienced competition from other plastics. One of these is polyolefin-based materials (Bani-Hani et al. 1999; Kopchik and Mein 2002; Rahman and Brazel 2004). Polyolefins include several important classes of polymers and their copolymers (Garbassi et al. 1995). Polyolefin-polyacrylate elastomer is one application for floor coverings (Lampinen 1999). In practice some polyolefin floor coverings have been found to be difficult to maintain in good condition after the PUR coating has worn (Hloch et al. 2003). This is due to their structure: their surfaces are non-polar and their adhesion properties are poor (Garbassi et. al. 1995). Bani-Hani et al. (1999) compared calcium carbonate –filled polyolefin plastomers (POP) to PVC-based formulations and concluded that POP-composites are very favourable. One example of

novel material which contains neither PVC nor plasticizer is a plastic composite tile with a wear layer of thermoplastic polymer, Enomer™ (Upofloor Oy 2005).

2.1.2 Self-cleaning surfaces

The modification of plastics for development of more self-cleaning surface materials is currently an important topic in many research groups. One natural self-cleaning surface is the superhydrophobic lotus leaf, from which water droplets roll off and wash off dust and soil (Zhang et al. 2005 and references therein). The main characteristic of these superhydrophobic surfaces is their roughness on the micro- and nanometer scale (Fürstner et al. 2005). Another mechanism of self-cleaning surfaces utilizes the photocatalytic and superhydrophilic properties of titanium dioxide (TiO_2) particles (e.g. Allen et al. 2004; Zhang et al. 2005). TiO_2 decomposes organic soil compounds photocatalytically using UV light and also in visible light. Some TiO_2 self-cleaning products such as tiles, glass and plastics have become commercially available.

2.2 NATURAL SOILS AND SOILING

The origin and content of natural soil on indoor surfaces varies due to the regional and local environment of the building and the activities taking place in the building (Thatcher and Layton 1995). The chemical composition of soils is very wide, and numerous substances may therefore be involved in the soiling of indoor surfaces. One of first analyses of soils was collected from pavements in six American cities 50 year ago. This 'street soil' contained the following components: water- and ether-soluble compounds, moisture, ash, silicate, iron, calcium and magnesium oxides, and nitrogen (Sanders and Lambert 1950). In addition, soil (in this context dust) contains several chemical compounds such as polycyclic aromatic hydrocarbons (PAHs) (Patrick 1999), volatile organic compounds (VOCs) (Wilkins et al. 1993; Franke et al. 1997; Wolkoff et al. 1998), lead (Lioy et al. 1998) and even the residues of cleaning agents such as fatty acid salts (Clausen et al. 1998) and linear alkylbenzenesulfonates (LAS) (Vejrup et al. 1999).

Furthermore, dust contains substances from biological sources such as gram-negative bacteria (Gyntelberg et al. 1994; Kildesø et al. 1998), endotoxins (Mølhave et al. 2000; Meyer et al. 2004), animal allergens such as dust mites, cat and dog allergens (Mølhave et al. 2000; Popplewell et al. 2000; Warner 2000), pollen (Fahlbusch et al. 2001), and fungi (Franke et al. 1997; Kildesø et al. 1998; Meyer et al. 2004; Arlian et al. 2001). Some of these components of dust are well known to cause adverse health effects.

A commonly encountered oily soil on surfaces in accessible areas originates from fingerprints. Sebum is one of the most complex mixtures of widely varying composition. The main components of aged sebum, with their representative levels, are triglycerides (32 %), free fatty acids (25 %), mono- and diglycerides (12 %), waxes (18 %), squalene (9 %) and cholesterol (4 %) and smaller amounts of other sterols and phospholipids (Schott 1972a).

The transport routes of soil are direct transfer and spillage or track-in with shoes, and deposition from air (Schneider 1995). After the soil has been deposited on surfaces, especially floor coverings may be subjected to severe stresses during normal traffic, which enhances the absorption of soiling agent. If there are particles of grit under the foot, the increased pressure produces microscopic corona stains, with the result that smaller particles have proportionately a much greater influence on stain uptake than large particles (Sears and Darby 1982).

2.3 EFFECT OF CLEANING

There are two kinds of studies concerning cleaning of surfaces in non-industrial indoor environments: one focuses on dust removal and its effects on indoor air quality resulting in health effects and the other on the efficiency of cleaning methods or the effects of method parameters on cleanability.

Airborne particle concentrations and human symptoms are affected by the type of surface material (Skov et al. 1990), the efficiency of cleaning in general (Roys et al. 1993; Skulberg et al.1999), cleaning frequency in offices (Sundell 1994; Stenberg et al. 1995;

Skyberg et al. 2003) and in schools (Wålinder et al. 1999; Smedje and Norbäck 2001), the efficiency of the cleaning method and the area of surfaces cleaned (Franke et al. 1997).

The efficiencies of individual cleaning methods differ significantly (Kildesø et al. 1998). Dry methods have been demonstrated to be effective in dust removal (Schneider et al. 1994a). In the literature, cleaning methods such as damp, slightly damp, semi-dry and wet methods are seldom defined exactly. An exception is the study of Vesley et al. (1987), who found that a slightly damp method (moisture 10% by dry mop weight) and dry (3% by dry mop weight) followed by wet mopping cleaned surfaces equally. Of the wiping materials, microfibre cloths have been found to be effective in soil removal both in damp and dry conditions (Nilsen et al. 1997, 2002). On the other hand, although wet methods have proved to be efficient in removing organic soil from surfaces (Andersen et al. 1997), they are disadvantageous because wet mopping of floors increases the airborne concentrations of viable bacteria and VOCs (Andersen et al. 1997; Smedje and Norbäck 2001).

When considering the impact of cleaning on soil removal in more detail, the physical and chemical parameters of different methods are taken into account. In hard surface cleaning mechanical action, such as pressure and speed, is essential (AORN 1998; Martens et al. 1999). For example, the method combination including dust mopping, machine buffing and renewed dust mopping has been found to be more effective than dust mopping followed by wet mopping (Schmidt et al. 1984). Similarly, the efficiency of mechanical action is increased by the coarseness of the cloth when removing oily soil from floor coverings (Duisterwinkel and Smits 1997; Schroeder et al. 2001). However, in many cleaning situations, the impact of mechanical action of cleaning cloths is related to surfactant concentration (Martens 2001).

2.4 ARTIFICIAL SOILING AND CLEANING

2.4.1 Model soils

Artificial soils are termed as model or standard soils, which are tailored to particular applications (Adamson and Gast 1997), e.g. for soil – surface material combinations such as the iron oxide pigment – linoleum (ASTM D4488, 2001). The artificial soils of hard surfaces may contain a single component (e.g. oxides, fatty acids) or several components (e.g. humus, house dust, sand, clay, minerals, oxides, carbon black, oils, fatty acids and solvents) (Pesonen-Leinonen 2003; Suontamo 2004). Soil mixtures mimic natural soils that appear on surfaces in a specific environment.

Fatty acids and triglycerides are used as model soils of natural oils and sebum. The model soils have included triolein, tripalmitin and palmitic acid in detergency studies (Engström and Bäckström 1987; Bäckström et al. 1988) or in soiling studies (Tikka et al. 2004). Oxides such as Fe_2O_3 (Ohlson and Wäänänen 1971) or Cr_2O_3 (Jokelainen et al.1976a, 1976b, 1979, 1982) alone and with an oily component, or single elements such as gold (Au) (Horowitz and Lindahl 1971) have also been used in cleanability studies in which the soil contains radioactive isotopes. The radioactive isotope tracers can be gamma-ray (e.g. ^{51}Cr) or beta-ray (e.g. ^{14}C) emitters. They are chemically bonded to the soil (Ohlson and Wäänänen 1971). The gamma-ray emitters are favourable as they detect soil embedded in the surface material. The tracers can also be e.g. a fluorescent oil-soluble dyestuff (Duisterwinkel and Smits 1997), carbon (Martens 2001) or even certain allergens (Jerrim et al. 2002).

2.4.2 Soiling and cleaning apparatus

For cleanability studies various kinds of laboratory scale soiling and cleaning apparatus have been developed to simulate and control soiling and cleaning of surfaces (Table 1). The first soiling apparatus were for textile floors, such as the Lisson apparatus standardised in the 1960s in Sweden. Similar applications have also been developed for hard floors by BCTC-CAMRASO (British Carpet Technical Centre and Cleaning and Maintenance Research and Services Organisation) (Burrows 1999), and established

standards include the Kappasoil method (EN 11378-1, 2000) and the application of the Hexapod drum method (EN 11378-2, 2001) for resilient floor surfaces (EN 14565, 2004).

The cleaning procedure can be performed manually (Nilsen et al. 2002) or instrumentally using various types of apparatus such as the Gardner (Hloch et al. 1995), Braive washability tester (Martens et al. 1999), Erichsen Washability Scrubbing Resistance Tester (Kuisma et al. 2003; Redsven et al. 2003), or a cleaning simulator (Kemppainen et al. 2002; Suontamo 2004). All these equipments offer a fixed method to study cleanability repeatably. However, the different types of apparatus differ in their soil distribution and cleaning mechanisms.

Table 1. Soiling and cleaning apparatus used in cleanability studies.

Apparatus	Area		Type of material		Type of soil		Standard	References
	Soiling	Cleaning	Carpets	Hard surfaces	Dry, particles	Sticky or liquid		
Gardner Washability Tester	x	x	-	x	x	x	ASTM D4488	Hloch et al. 1995; Garvens and Krüssmann 1997
Erichsen Washability Tester	x	x	-	x	x	x	ASTM D2486, ASTM D3450 EN 11998	Nilsen et al. 2002, Kuisma et al. 2003
Sheen Washability Tester	x	x	-	x	?	?	ASTM D2486 , ASTM D4828	Hloch et al. 2003
Braive	?	x	-	x	x	x	ASTM D 2486	Martens et al. 1999, Martens 2001
Kappasoil	x	-	x	x	x	-	EN 11378-1	Burrows 1999
Cleaning simulator	x	x	-	x	-	x	-	Suontamo 2004
Soiling Drum Apparatus	x	-	x	x	x	-	EN 14565, EN 11378-2	Redsven et al. 2003
Test chamber and apparatus for dusting	(x)	x	-	-	x	?	-	Jerrim et al. 2002
Spin coater	x	-	-	x	-	x (liquid)	-	Tikka et al. 2004
Rolling perforate cylinder (RPC)	x	-	x	?	x	-	- *)	Lewis et al. 1999

x = applicable, — = not applicable, ?= not defined
*) not standardized but validated

2.5 SOIL ADHESION

After the soil has been transported to the surface material the adhesion follows. Many theories of adhesion have been proposed, which together are both complementary and contradictory (Lipatov 1995; Shultz and Nardin 1999; Petrie 2000). The theories include 1) mechanical interlocking, 2) adsorption (thermodynamic) theory, 3) electrostatic and diffusion theory, and 4) chemical bonding theory. Mechanical interlocking is associated with soil adhesion and detachment (Short 1972; Michalski et al. 1998a). It usually depends on the roughness of the surface (Garbassi et al. 1995). Roughness aids adhesion by increasing the total contact area between soil and surface material (Petrie 2000). However, particulate soil can adhere on smooth plastic surfaces (Kissa 1987 and references therein; Garbassi et al. 1995). Particles adhere on substrates in the region of the adhesion zone, which affects the surface geometry of particles and substrate and their elastic and plastic properties (Lange 1972; Rimai and DeMejo 1996). In addition, particles may adhere to surfaces as a result of capillary condensation (Israelachvili 2002). Thus, e.g. an oil film on a surface captures soil particles by adsorption (Kissa 1987), i.e. liquid soiling enhances particle soiling. When the soiling agent is liquid, wettability of a surface is strictly correlated with adhesion (Garbassi et al. 1995) according to adsorption theory. Further, electrostatic adhesion of particulate soil is due to an electrical double layer or electrostatic forces (electric charge) (Garbassi et al. 1995; Ziskind et al. 1995). Chemical adhesion between two materials enhances the level of adhesion by chemical bonds such as ionic bonds (Shultz and Nardin 1999). After soil has become adhered on surfaces it should be removed to fulfil the cleanliness requirements of the surface. The removal energy needed for cleaning must overcome the total adhesion forces (Kohli 2002).

2.6 CONTACT ANGLE AND SURFACE FREE ENERGY

Contact angle, θ, is defined as the angle formed between the liquid-vapour and the liquid-solid interfaces, at the solid-liquid-vapour three-phase contact line (Lam et al. 2001b). Contact angle is also a measure of the hydrophobicity and wettability of a surface. It can provide information about surface energies, surface heterogeneity and surface roughness (Lam et al. 2001b), as well as about self-cleaning properties of coatings (Zhang et al. 2005). Contact angle measurements are one of the most sensitive methods providing information on the outermost polymer surface of a few angstroms (Garbassi et al. 1995; Weikart et al. 1999; Kato et al. 2003). Numerous techniques are currently available for measuring contact angle. The most commonly used method is the contact angle for a drop of liquid resting on a horizontal solid surface (a sessile drop) (Adamson and Gast 1997). A drop profile is determined using the conventional goniometer-telescope or the more advanced axisymmetric drop shape analysis – profile (ADSA-P). ADSA-P employs a strategy that a fits the shape of an experimental drop to a theoretical drop profile according to the Laplace equation of capillarity (Kwok et al. 1997). The measured contact angles are higher or lower than the equilibrium contact angle on the practical, rough and heterogeneous surfaces. Two relative reproducible contact angles are the largest advancing (θ_A) and the smallest receding (θ_R) contact angle (Grundke et al. 2003). Advancing and receding contact angles are measured from a sessile drop, as liquid is injected into or withdrawn from the drop slowly by means of syringe assembly (Lam et al. 2001b). Good and van Oss (1992, ref. Gindl et al. 2001) stated that if only one angle measurement is performed, the advancing angle should be studied. The contact angle is measured repeatedly on a fresh surface and time is not a factor. The only phenomena measured are the physical interactions between the liquid and the solid (Lugscheider and Bobzin 2001). Furthermore, the moving three-phase contact line may alleviate the effect of equilibrium spreading pressure, local irregularities, and defects on the surface (Lam et al. 2001b). The employment of advancing contact angle for the solid surface free energy determination is a common practice (van Oss et al. 1988; Adamson and Gast 1997; Lam et al. 2001a).

Contact angle measurements are most widely employed to estimate solid surface free energy. Sharma and Rao (2002) listed the main approaches for the estimation of surface free energy such as that of the Zisman (1952), the Fowkes (1964), Owens and Wendt

(1969) geometric mean approach, Wu (1971) harmonic mean, van Oss et al. (1986) the acid-base approach, and the equation of state approach derived by Neumann et al. (1974) and Li and Neumann (1992). The Owens and Wendt approach (also known as the Owens-Wendt-Rabel-Kaelbe model) is commonly used to estimate surface free energies of complex surfaces (Castilho Pereira et al. 1993; Jacobasch et al. 1993). It is efficient for comparison of various surfaces, which is important for practical applications (Michalski et al. 1998b).

2.7 DETERMINATION OF CLEANABILITY

Numerous methods are available to determine surface cleanability, ranging from subjective visual techniques to molecular scale techniques. In practical situations, the method selected is based on the cleanliness level required, on the type of surface material and on the type of soil (Chawla 2001). Relevant techniques and their main features from the viewpoint of this study are described briefly in the following.

2.7.1 Colorimetry

Cleanability is commonly determined as the colour change of the soiled or cleaned surface material. Colorimeters measure the light reflected from an object using three sensors which are filtered to have the same sensitivity colour-matching functions of red, green and blue as the human eye. Then, the tristimulus values measured from the spectral reflectance data are calculated by performing integration (Minolta 1998). Colours are specified using the CIELAB colour space (CIE, Commission Internationale de l'Eclairage). The coordinate L* denotes colour lightness, a* denotes hue on a red-green axis and b* denotes hue on a yellow-blue axis. An integral standard light source enables the standardization of measurements even when ambient light is inadequate (Barret 2002).

Colorimeters have been employed to assess the degree of cleaning or soiling or cleaning efficiency. Hue or merely lightness of colour is quantified using the readings of L*, a* and

b* values or of L* values alone. The results are commonly expressed as cleaning efficiency (cleaning index) based on lightness L* values measured from original, soiled and cleaned surfaces of a specimen (Krüssmann and Garvens 1997; Burrows 1999; Kuisma et al. 2003; Redsven et al. 2003). L* values are employed by assessing the soiling rate of various surface materials such as stones (Thornbush and Viles 2004). Stain release and adhesion can also be evaluated in terms of ΔL or the colour difference in terms of ΔE (Bohnert et al. 1998; Pitts et al. 1998).

2.7.2 Radiochemical method

The first cleaning technological application of radiochemical method was to evaluate soil removal in detergent tests, e.g. as adsorption of surfactants onto fabrics and as soil adhesion on surfaces (Cramer 1972; Shebs 1987), or the efficiency of surfactants to remove solid organic soils from hard surfaces (Engeström and Bäckström 1987). A radiochemical method is based on the measurement of the amount of radiolabelled soil on surface materials. Its excellent sensitivity permits accurate measurements at very low levels of soil (Cramer 1972). In the 1970s the method was utilized in cleanability studies of surface materials such as carpets, PVC-floorings and linoleums (Horowitz and Lindahl 1971, Ohlson and Wäänänen 1971; Jokelainen et al. 1976a, 1976b, 1979 and 1982). Both gamma-ray emitters (^{51}Cr, ^{59}Fe, ^{56}Mn) and beta-ray emitters (^{3}H, ^{14}C) have been employed as radioactive tracers. An advantage of the use of gamma-ray emitters is that their use is non-destructive and therefore the accumulation or removal of soil can be followed phase by phase.

The radiochemical method has been used in various cleaning technological applications and in various experimental designs. For example, the rotating disk technique with radioactively labelled solutes has been used to measure the solubilization rates of pure, solid fatty acids and solid monoglycerides in several surfactant solutions (Chen et al. 1997). The effectiveness of cleaning methods on metal compounds has been evaluated using radioactive tracers in semiconductor manufacturing process (Wang et al. 2001). Bacterial adhesion on furanone-coated materials has been determined using radioactivity and the triated thymidine ‘adhesion assay’ (Baveja et al. 2004). Deuterium oxide (D_2O)

has been utilized to study the mechanism and interaction of water and a model organic contaminant (isopropyl alcohol in semiconductor processing) with dielectric oxides (Raghu et al. 2004). One application of surface analysis methods based on radiochemistry is MESERAN (measurement and evaluation of surfaces by evaporative rate analysis), which uses a thin-end-window Geiger Müller to detect molecules of a volatile compound containing ^{14}C (Benkovich and Anderson 2003; Chawla 2003). The radiochemical method is employed in many medical, environmental, industrial, space, scientific, and national security applications (Franks et al. 1999).

2.7.3 Contact angle measurements

Contact angle measurements are employed in precision cleaning (e.g. in semiconductor industries) to detect soil on surfaces. The measurements are based on different hydrophobic-hydrophilic characteristics of soil and solid surfaces (Chawla 2001). The presence of soils such as hydrocarbons (oils) on hydrophilic surfaces, protein film but not heterogeneous milk film on very hydrophilic or very hydrophobic materials are detected using the sessile drop technique (Yang et al. 1991 and references therein). The quantity of adsorbed soil on surfaces cannot be determined quantitatively but has been used as an index of surface cleanliness (Yang et al. 1991). Consequently, the contact angle measurement is a pass/fail test to determine the need for recleaning or for more critical analysis (Geosling and Koran 2001). Further, the dynamic contact angle analysis has been employed to evaluate the kinetics of protein removal from various hydrophobic and hydrophilic surfaces using the applied Wilhelmy plate technique (Davies et al. 1996).

Sessile drop technique has been used for surface characterisation to predict soil (food particle) behaviour on plastic surfaces (McGuire and Kirtley 1988). The authors suggested that there might be relationship with the polar component of surface free energy. More recently, Garvens and Krüssmann (1997) studied the relationship between contact angle and the soiling tendency and cleanability of resilient flooring materials. They also examined the feasibility of determining the adequate number of polymeric floor polish layers for good cleanability and estimated optimum cleaning costs by contact angles.

2.7.4 Foil sampling and optical detection

This method has been used in cleanability studies to assess the cleaning efficiencies of different methods (Schneider et al. 1994a; Nilsen et al. 2002), to examine dust build-up on surfaces (Kildesø et al. 1999) or in intervention studies (Kildesø et al. 1998). The method has also formed the basis for proposing a sampling strategy and surface dust limits in relation to cleaning and the quality of the indoor environment (Schneider et al. 1994b). Sampling is employed to identify or to determine the concentration of a substance or substances on a limited surface area (Godish 2001). Lioy et al. (2002) compiled sampling methods. Common sampling methods include sticky sampling, wiping and vacuuming (Kildesø and Schneider 2001). Soil such as dust and other particulate matter on surfaces is collected using adhesive and transparent gelatine foils and is quantified by an optical method, which is based on laser light extinction (Schneider et al. 1996). The lower level of detection for dust on an object is less than 0.5 % of surface covered by dust (Schneider et al. 1996). Sticky samplers are capable of giving semi-quantitative and quantitative results because they have high collection efficiency (stickiness of surface) and wide application (applicable for measuring particulate matter using microscopy or image analysis (Wheeler and Standcliffe 1998; Yoon and Brimlecombe 2001). Another advantage is that the gelatine foils follow minor curvatures of surfaces (Schneider 1994).

3 AIMS OF THE STUDY

The purpose of the present study was to examine the feasibility of cleanability determination methods and to investigate the cleanability of plastic materials from the molecular scale (III) to practical scale (V).

The specific aims were:

1. To examine the feasibility of a colorimetric method in cleanability studies of plastic materials (I, II, IV).

2. To establish a radiochemical method to investigate soil accumulation on PVC model surfaces, and to apply the method to investigate the effect of composition of PVC model surfaces on cleanability (III).

3. To examine the feasibility of a contact angle measurement technique to characterize plastic materials and to predict their cleanability (I, II).

4. To study the suitability of foil sampling and an optical method to detect soil (dust) accumulation on surfaces, and to evaluate the effect of cleaning in practical office environments (V).

4 MATERIALS AND METHODS

4.1 Study design

The cleanability of PVC model materials and of commercial plastic materials was studied using three soil determination techniques. In addition, the contact angle measurement technique was employed to characterize the commercial plastic materials (Figure 1).

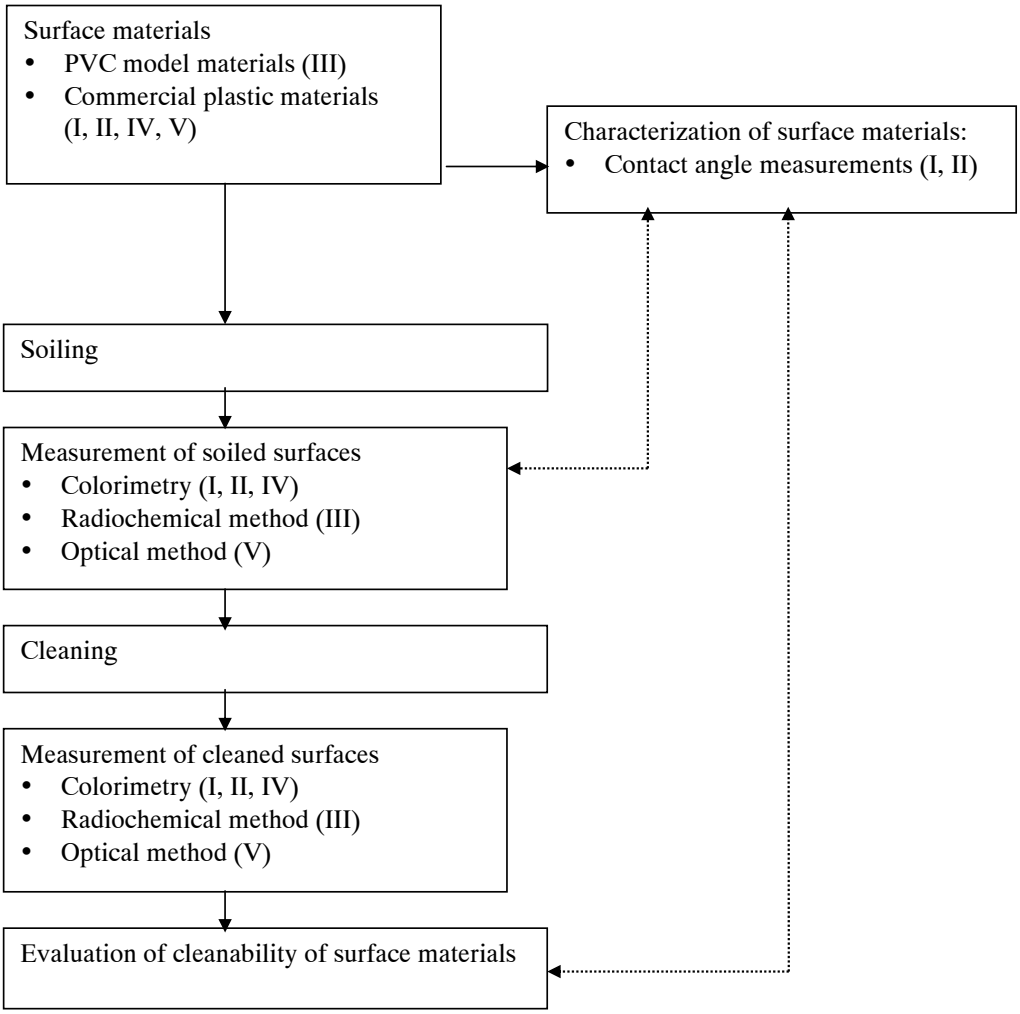

Figure 1. The phases of the study.

4.2 Surface materials

PVC model materials (III) and light-coloured commercial plastic flooring materials (I, II, IV, V) were used (Table 2). Detailed descriptions of the materials are found in Table 1 of Papers I, II and III. The PVC model material contained either DOP (dioctyl phthalate) or Hexamoll DINCH (di-isononyl-cyclohexane-1,2-dicarboxylate) as plasticizer. The commercial flooring materials were selected to represent the most used ones according to the information of retailers or the manufacturers as follows: PVC flooring materials, PUR-treated PVC flooring materials, and PUR-treated thermoplastic polymer material.

Table 2. Surface materials used. Detailed information of materials is presented in Table 1 of Papers I, II and III.

Materials	Top layer	I	II	III	IV	V*)
PVC model materials	Bulk	-	-	x	-	-
PVC flooring materials, sheet	Bulk	x	x	-	-	x
	PUR-treated	x	x	-	x	x
PVC composite tile	PUR-treated	x	x	x	-	x
Thermoplastic polymer	PUR-treated	x	x	-	-	-
Other flooring materials	Various	-	-	x	-	x

*) PVC materials and some linoleums were maintained using polymer floor finish

4.3 Soils

Model soils such as particle soil (I, II) and oily soils (I, II, V) were used as soiling agents. They were multi-component model soils simulating practical soils on indoor surfaces such as in corridors near the front door or stubborn oily soils in offices in hard-soiling environments. A simple model soil labelled with radioactive isotope ^{51}Cr was used to monitor soil accumulation (III). Detailed descriptions of the soils are found in Table 2 of Paper I, II and III, and in Table 4 of Paper IV. In Paper V, dust occurring naturally in office environments was as soil.

4.4 Soiling and cleaning methods

Soiling was performed by three techniques: 1) soil was applied as a suspension with a pipette, allowed to dry and fixed by scrubbing (III), 2) soil was deposited with a pipette, spread with the Erichsen Washability Tester and allowed to dry (I, II, IV), or 3) soil was applied using the Soiling Drum Tester including fixing with Hexapod (I, II). Soils were removed by a straight-line wiping movement using the Erichsen Washability Tester (I, II, IV) or by an elliptical movement using the pilot apparatus, Mini Cleanability Tester (III). In the studies, water and model detergents (Table 3 of Paper I and II) and commercial detergents (Table 3 of Paper IV) were used as cleaning solutions, and a microfibre cloth with the texture presented in Figures 1 and 2 of Paper IV was used as cleaning equipment.

In paper V, the naturally settled soil on surfaces contained dust particles of the office environment. Surfaces were cleaned with practical cleaning methods. Detailed descriptions of soiling, cleaning methods and cleaning solutions can be found in the original publications.

4.5 Determination of cleanability

Cleanability was determined using the radiotracer technique (III), colorimetry (I, II, IV) and an optical method (V). Surface properties were characterized using the contact angle measurement technique (I, II). The methods are compiled in Table 3. Detailed descriptions of the methods are presented in the original Papers I, II, III, IV and V.

The contact angles were measured on flooring materials using an optical contact angle meter CAM 100. The meter determines contact angles by fitting the Laplace equation to the shape of the axisymmetric menisci of a drop. Two different techniques were employed: the static contact angle, θ_S, and the advancing contact angle, θ_A, techniques. The contact angles measured were used to determine solid surface free energy, γ_S, and its polar and dispersion component with the Owens and Wendt approach (1969).

Table 3. Methods used for cleanability determination and also for surface characterization. The number of measurements presented in the last column includes the following factors: contact angle (θ_A), the number of study materials (m), replicates of each material (r), number of cleaning solutions (s), number of soiling and cleaning times (t), the number measurements of each replicate (n) and the number of cleaning method (c).

Paper	Determination of cleanability or surface property	Apparatus and manufacturer	Result	Measurements
I, II	Contact angle measurement	CAM 100, KSV Instruments Ltd, Finland	Surface properties related to cleanability	10m * 4 probe liquids * approximately 5 drop * approximately 24 θ_A = 4800 measurements
III	Radiochemical tracer method	Detector, a Bicron 2"x2" NaI(Tl)-crystal Bicron Corporation, Ohio, USA Multi-channel analyzer Canberra Inc. Meriden, USA	Proportional soil residue	6m*6r*6t*1s*2n = 432 measurements
I, II, IV	Colorimetric method	Chroma Meter CR-210, Minolta Co, Ltd, Japan	Cleaning index, soil residue, total soiling	10m*5r*1t*3s*15n = 2250 colour measurements (I, II) 12m*5r*1t*4s*2c*15n = 7200 colour measurements (IV)
V	Foil sampling and optical method	BM Dustdetector, BM Environmetal Engineering, the Netherlands	Proportional dust coverage area	Approximately 3100 measurements

The colorimetric method was used to detect black-coloured soils on the light-coloured surface materials. Data from the colorimeter was quantified using the L* scale of the CIELAB colour scale. The colorimeter was calibrated each time it was taken in use. The rates of soiling and cleaning were calculated using the equations presented in Table 5 of Paper II. The result was the average of five individual readings of five replicates on each flooring material.

The radiochemical method was employed to detect soil labelled with radioisotope ^{51}Cr. The activity was measured using a gamma–ray scintillation detector assembly. The counting system consisted of an NaI(Tl) detector optically mounted to the photomultiplier tube, which was coupled with a multi-channel analyzer. The detection window of the

spectrum of ^{51}Cr and the energy summit channel was determined daily. In addition, the detector was calibrated daily using a reference sample of activated chromium. The counts were recorded for five minutes (cp5m). The counting geometry was made constant using the special sample frame. The result was the average of six replicates.

In the field study (V), the accumulation of dust particles on surfaces was determined using sampling with transparent gelatine foils followed by the use of an optical method based on light extinction. Dust was collected on the surfaces of furniture and floors in randomly selected office rooms in six buildings. The instrument was calibrated with black slides daily (Schneider et al. 1996). Dust amount was expressed as a percentage of the surface area covered by dust particles.

4.6 Statistical analyses

Multiple linear regression analysis was employed to examine the relative contributions of the surface properties in terms of contact angle, surface free energy and its polar and dispersion component, and to the cleanability of flooring materials (I, II). A stepwise procedure was used to select the independent variable that should have been included in the model to predict cleanability. A variable was progressively entered into the model if the significance level of its statistical score was lower than 0.05 and removed if the significance level was lower than 0.01. Evaluation of the validity of the multiple regression analysis was confirmed by consideration of residuals and by the use of an ANOVA (Norusis 2004). The effect of cleaning, building and type of surface on dust concentration was analysed as an unbalanced factorial experiment by analysis of variance (V). For all analyses the level of statistical significance was set at 0.05. Statistical analyses were performed using SPSS version 12.0. (SPSS Inc., Chicago IL, USA), except that analysis of variance and principal component analysis of Paper V were conducted with SAS version 6.12 (SAS Institute Inc., Cary, NC, USA).

5 RESULTS

5.1 Cleanability determined using colorimetry

Colorimetry was used to assess changes in the amount of soil on the commercial plastic flooring materials after soiling and cleaning. The determination of a transition was performed on the basis of lightness L* values of CIELAB and was used to obtain the parameters of cleanability (Table 5 of Paper II). As shown in Figure 2 the plastic flooring materials were soiled differently and also cleaned differently.

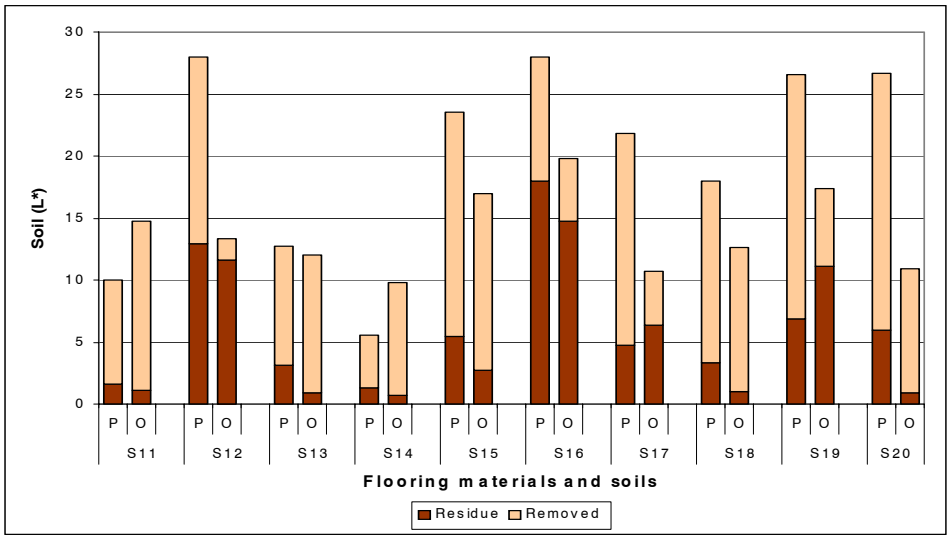

Figure 2. Soiling of particle (P) and oil (O) soil (whole bar) and the share of soil removed (light bar) and soil residues (dark bar) on flooring materials. The materials were cleaned with a weakly alkaline model detergent. The results are expressed as means of five replicates. The error bars are presented in Figures 1 and 2 of Paper II. The descriptions of flooring material are presented in Table 1 of Papers I and II, and the compositions of soil are presented in Table 2 of Papers I and II.

Colorimetric measurements (I, II) had good repeatability. Standard errors (SE) of L* values were <0.6 L*value for the measurements obtained from original colour, particle- and oil-soiled surfaces, and from cleaned surfaces (I, II). In addition, the possible relationship between the original colour and the cleaning indices and soil residues was investigated. There was no significant correlation between the original colours of

materials and the cleaning indices of particle- and oil-soiled surfaces (p=0.140 and 0.175, respectively) (I, II), or of soot-oil soiled surfaces (p=0.212) (IV). On the other hand, soil residues of particle soil had a weak correlation with the original colours of materials (r=0.679, p=0.031). As a result, the colour measurements were reproducible with acceptable standard errors, and could therefore be used to examine overall cleanability of materials. However, the cleanability results are comparable only when they are obtained from the same study system.

5.2 Radiochemical method to determine cleanability

The radiochemical method including a pilot apparatus for cleaning, the Mini Cleanability Tester, was established to determine the soils labelled with radioactive isotope ^{51}Cr. The procedure of the method was developed to examine the accumulation of model soils on the PVC model materials (containing either DOP or Hexamoll DINCH plasticizer) and on vinyl composite and linoleum flooring materials during six successive soiling-cleaning cycles.

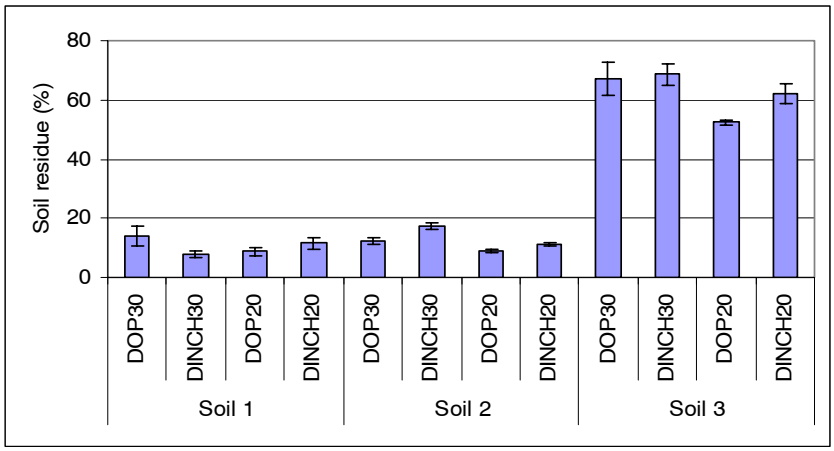

Figure 3. The average accumulation of soils on PVC model surfaces after the sixth soiling-cleaning cycle determined with the radiochemical method. The error bars are standard errors of the mean soil residue-%. Soil 1 = chromium oxide, Soil 2 = chromium oxide + triolein, and Soil 3 = chromium acetyl acetonate + triolein. Plasticizers: DOP = dioctyl phthalate and DINCH = Hexamoll DINCH, di-isononyl-cyclohexane-1,2-dicarboxylate. The values 20 or 30 refer to the concentration of plasticizers as wt%.

The accumulation of model soils on PVC model materials and flooring materials after each soiling-cleaning cycle is presented in Figure 1 (a-f) of Paper III. The accumulation of Soil 1 (chromium oxide) showed no increase on PVC model materials and flooring materials over the six soiling-cleaning cycles. Soil 2 (chromium oxide + triolein) accumulated only on linoleum, soil residue increased 22 %-units. Soil 3 (chromium acetyl acetonate + triolein) showed increasing accumulation from the first soiling-cleaning cycle to the sixth cycle on all surfaces. The soil residue increased 42 %-units on PVC1, 55 %-units on PVC2, from 26 %-units on PVC3, from 17 %-units on PVC4 and from 48 %-units on linoleum. The results of vinyl material were rejected because Soil 3 adhered to some extent on the sides of the discs during the cleaning phase. Figure 3 presents the accumulation results after the 6th soiling-cleaning cycle on PVC model materials: both type and amount of plasticizer affected the accumulation of the soils with triolein (Soils 2 and 3). Consequently, a reproducible radiochemical method, which included a pilot cleaning apparatus, was established to provide information of the accumulation of soils on plastic materials.

5.3 Contact angle measurements to predict cleanability

5.3.1 Contact angle measurements and surface free energies

The feasibility of contact angle measurements on plastic materials was examined using a drop expanding technique. The advancing contact angles θ_A obtained with the polar liquids (distilled water, ethylene glycol, formamide) and for the apolar liquid (di-iodomethane) on the commercial plastic flooring materials (S11-S20) are presented in Table 4 of Paper I. The contact angles θ_A of probe liquids on the materials were correlated ($r = 0.84$, $p<0.01$). The highest water contact angles θ_A, 87° and 115°, were measured on PVC flooring materials S12 and S16, respectively. The contact angle θ_A was at least 11° lower on PUR-treated PVC flooring material than on PVC flooring material. The values of contact angle θ_A varied over a certain range (Figure 1 of Paper I).

Surface free energy (SFE) of flooring materials and its polar and dispersion components are presented in Figure 3 of Paper II. The highest SFE was calculated for PUR-treated flooring material S20 (49 mJ/m^2) and the lowest for PVC flooring material S16 (26 mJ/m^2). The polar component of SFE was lowest for PVC flooring materials S12 and S16 (> 0.5 mJ/m^2) and for the PUR-treated flooring material, ranging from 3.6 mJ/m^2 to 18 mJ/m^2.

The drop expanding technique applied on commercial plastic materials proved to be reproducible. Standard errors of contact angle measurements were generally less than ±1.6°. Consequently, the surface properties provided by contact angle measurements could be used to evaluate cleanability.

5.3.2 Cleanability related to contact angles and surface free energies

In order to evaluate the feasibility of contact angles for prediction of cleanability of plastic materials, the relationship between cleanability and the surface properties in terms of water contact angle (wettability), surface free energy and its polar and dispersion components was examined. Cleanability was expressed as cleaning indices obtained from the particle- and oil-soiled flooring materials (Figure 4). The water contact angle θ_A had a negative correlation with the cleaning index of particle-soiled surfaces and a weak correlation with the cleaning index of oil-soiled surfaces (Figure 3 of Paper I). Both surface free energy (SFE) and the polar component of SFE were related to the cleaning indices of the particle-soiled flooring materials and to contact angle (Figure 4 of Paper I). Thus, the lower the contact angle and the higher SFE or the polar component of SFE, the higher was the cleaning index. There was no association between the contact angles of the polar liquids and total soiling caused by particle or oil soil.

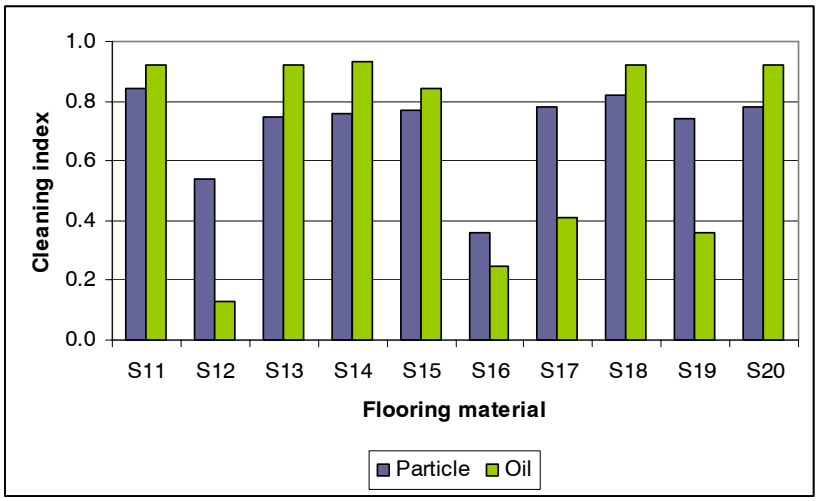

Figure 4. Cleaning indices of flooring materials soiled with particle soil and oil soil. Weakly alkaline model detergent served as a cleaning agent. The descriptions of flooring materials and codes are presented in Table 1 of Papers I and II.

Multiple linear regression analysis was employed to determine the potential of surface properties obtained with contact angle measurements to predict cleanability. For the cleanability of particle-soiled flooring materials, the regression was a good fit (regression coefficient: $R^2 = 0.88$), and the overall relationship was significant ($p=0.03$). Contact angle explained 75 % ($p<0.001$). For the cleanability of oil-soiled flooring materials, the regression was a good fit (R^2 = 76 %), and the overall relationship was significant ($p=0.019$). Contact angle explained 44 % and SFE increased R^2 by 32 % ($p<0.05$). The results suggested that the wettability of flooring materials would explain the cleanability of particle- and oil-soiled surfaces.

5.4 Foil sampling and optical detection

The suitability of the foil sampling method and the optical method to detect dust accumulation was studied in six office buildings (A-F) (Table 1 of Paper V). Dust accumulation on furniture and floor surfaces during one week and the effect of cleaning were measured as the percentage of the surface area covered by dust particles (Figures 1-3 of Paper V and Figure 5).

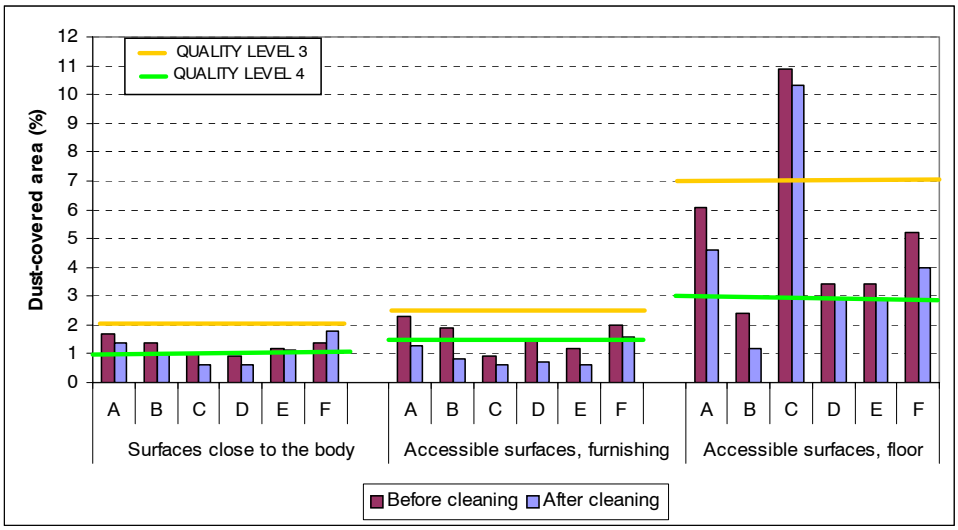

Figure 5. The accumulation of dust on different surfaces in the buildings before and after cleaning expressed as dust-covered area (%). The green line refers to the quality level 4 (second best) and the yellow line to level 3 (third best) defined by INSTA 800 (2003). Letters A-F are codes for the buildings. Detailed information on the buildings is presented in Table 1 of Paper V.

Dust accumulation was found to be heaviest on the 'not accessible surfaces' (e.g. shelves above 180 cm from the floor) and the 'surfaces close to the person' had generally least dust. However, the cleaning failed to reduce the dust amount on the surface 'close to the person' in the building F. The condition of the surface of flooring materials influenced the dust amount, which was lowest on the well-finished and polished floor covering in building B, compared to the flooring materials with maintenance problems in building C.

These results were ranked on the basis of the cleaning quality levels of the INSTA 800 (2003) standard, in which the best quality level is level 5 and the poorest is level 1. The surfaces were ranked according to the evaluation procedure, which takes into account the scattering range of dust measurements. The results showed that in buildings C and D the dust level of furniture surfaces was at the best levels 5 or 4 between cleaning times. Cleaning removed dust, but not enough to reach the best quality levels on all surfaces of the buildings. The results indicated that each office building (A-F) had its own quality profile measured as proportional dust covering. The dust measurement procedure proved to be suitable to detect the differences between dust levels on the office surfaces and the effect of cleaning.

6 DISCUSSION

6.1 Colorimetry to determine cleanability

The results indicated that colorimetry is a feasible method to estimate the cleanability of flooring materials, especially when using the soiling procedure of particle soil described in EN 14565 (2004). This is due to the fact that the excess soil, loose and mainly larger particles resting on surfaces, is removed by vacuuming according to the soiling procedure of the standard. Changes in the colour of the soiled surfaces are detectable with the colorimeter, shown in Figure 2. The use of a template in the colour measurements ensures that the values obtained before cleaning, after cleaning and after soiling were from exactly the same locations of each specimen. It was necessary to use dark soil on light-coloured surfaces in order to make the initial difference between soil and surface as great as possible. Thus, the colour and the amount of soil can be considered to be associated with each other.

Cleanability can be presented using cleaning indices and soil residues. Cleaning index expresses the proportional soil removal, indicating the effectives of the cleaning method but at the same the cleanability of a material, because the cleaning is performed with a similar procedure using similar cleaning parameters, and therefore the effect of the method can be excluded. On the other hand, soil residue could be considered as a measure of soil adhesion on a surface, and of the soil that cannot be removed using the given method. Both parameters are feasible. However, if cleanability of a surface material is considered to include soiling tendency, the cleaning index of cleanability parameters describes cleanability rather well. In addition, it should be taken into consideration that the original colour may possibly affect the value of soil residue if there are minor amounts of soil left. Nonetheless, in this study the soil residues and cleaning indices correlated strongly ($r=-956$, $p<0.0001$, $r=-951$, $p<0.0001$, respectively), indicating that both parameters are feasible to evaluate the cleanability of flooring materials.

Colorimetry provides a rapid, non-destructive, and quantitative measure of the colour of surfaces, confirming the conclusion of Pitts et al. (1998). It is simple to use and also

suitable for field studies as the area inspected is not limited. When colorimetry is compared to other determination methods, it quantifies the colour changes of surfaces, but the visual comparative ranking method or the use of the white-gray scales method (EN 20105-A02, 1994) provides the qualitative data. No panel groups as described in ASTM D 5343 (1997) or by Sears and Darby (1982) are needed. Secondly, using colorimetry the amount of knowledge needed to establish the experimental design is lower and cheaper compared to the radiochemical tracer technique. Colorimetry offers an easy to use and reproducible method to evaluate the cleanability of light-coloured flooring materials, as dark-coloured soil is used. In addition, it can take into account the fact that the results obtained are valid when the described study design is used.

6.2 Radiochemical method and effect of PVC composition on cleanability

6.2.1 Radiochemical method to detect soil accumulation

The radiochemical method established proved to be feasible for assessing soilability, cleanability and soil accumulation on plastic materials. Radiotracer technique was employed using ^{51}C as a radioactive isotope that is chemically bound to the soiling agent. The ^{51}Cr tracer has been used in cleanability studies to quantify soil adhesion to resilient flooring materials (Ohlson and Wäänänen 1971; Jokelainen and Uusi-Rauva 1976a). An advantage of ^{51}Cr as a gamma-ray emitter is that it can also detect soil embedded in the surface material. The amount of soil is proportional its radioactivity. The radiotracer technique is a non-destructive method which allows detection of soil accumulation after several soiling-cleaning cycles. Similarly, soil release could also be monitored with this technique.

As a part of the radiochemical method, a new pilot cleaning apparatus, Mini Cleanability Tester, was developed. The apparatus was modified especially for small disks made of plastic materials. The elliptical cleaning movement and the stepless adjustable cleaning

pressure distinguishes the Mini Cleanability Tester from the Erichsen Washability Tester, the cleaning movement of which is a straight line with two adjustable pressures. The cleaning of disks of plastic materials can be performed in a constant manner. The Mini Cleanability Tester would also be suitable for other small-sized materials and could be adapted for small-square samples.

In cleanability studies the amount of soil is presented quantitatively as a proportional soil residue as in the studies of Ohlson and Wäänänen (1971) or Jokelainen and Uusi-Rauva (1976a). Thus, using the soil residue in cleanability evaluations the soiling tendency of a material can be taken into account. In addition, the results obtained with the radiotracer technique could be shown as radioactive counts (Baveja et al. 2004) or in units of molecules cm^{-2} (Dobrozemsky 1995). The radiotracer technique can provide data at the molecular level. However, the inspection area is limited to the diameter of an NaI(Tl) crystal (of a photomultiplier tube), costs are relatively high and a high level of skill is required. A limitation of the study design established could be that triolein was not labelled and chromium (^{51}Cr) acetyl acetonate was assumed to describe triolein removal. Triolein could be labelled with carbon ^{14}C (Engström and Bäckstöm 1987) or with ^{3}H (Thompson 1994). However, in this study the measurement of a beta-ray-emitting isotope was not possible due to the different measuring technique.

By using the radiotracer technique in this kind of study design, the amount of soil labelled with radioactive isotope could be detected after each soiling-cleaning cycle. Thus, to understand the complexity of soiling and cleaning, simple model soils could be used. Similarly, the simple soil-substrate system could be used to determine the characteristics of the model soils which are related to cleanability.

6.2.2 Effect of PVC composition and soil on accumulation

In the radiochemical study both the composition of PVC model materials and the type of soil were associated with soil accumulation. The accumulation of Soil 1, chromium oxide as a dry powder soil, ended after the first soiling-cleaning cycle. The PVC surfaces appeared to be rather smooth, but there may have existed some crevices or other

unevenness (at the macroscopic scale) for particle soil to fill up. According to Compton and Hart in the 1950s in the absence of any oil, geometric bonding is the primary mechanism in particle soiling (Short 1972). In addition, pressure and mechanical action during soiling increase soil adhesion on plastic surfaces (Sears and Darby 1982; Kissa 1987). On the other hand, residues of clay powder have been shown to increase almost linearly with the plasticizer content of vinyl flooring (Sears and Darby 1982). The apparent disagreement between these results may be due to the fact that the soiling and cleaning procedures including soil characterisation (particle size and shape) were not reported. Furthermore, the soilability of PVC was evaluated visually with a ten-point scale (Sears and Darby 1982; Colletti et al. 1998).

If the soil contained an oily compound, triolein, the amount of plasticizer first affected the accumulation. Later after repeated soiling and cleaning, the type of plasticizer began to have an effect. Thus, increased soiling can be attributed to increasing softness of the PVC material. This agrees with the results according to which the softer compositions stained more than the harder ones, but at the same the soiling depended on the type of plasticizer (Sears and Darby 1982). Colletti et al. (1998) showed that soiling (staining) increased when the content of plasticizer exceeded 20 phr (parts per hundred parts of resin by weight). Similarly in this study, in which the compositions of plasticizers were 20 wt% and 30 wt%, the increase of oily soil residue was as expected. The presence of triolein slightly affected the residue of particle soil (chromium oxide), increasing the accumulation of Soil 2. A thin oily layer is known to affect the adhesion of particles to surfaces (Schott 1972b).

In this study, the effect of Hexamoll DINCH on the cleanability of PVC materials was investigated for the first time. The PVC materials with Hexamoll DINCH became more soiled than those with DOP. On the other hand, of the 19 plasticizers studied DOP has been reported to be the poorest in stain resistance (Sears and Darby 1982). This might indicate that Hexamoll DINCH would not be the first choice to plasticize PVC applications subjected to hard soiling. The soilability associated with plasticizers depends on the structure properties such as chain length, volatility, concentration, extraction resistance and solubility of the plasticizer (Colletti et al. 1998). On the other hand, each component of vinyl flooring affects soil resistance (Colletti et al. 1998). PVC flooring materials with more hydrophilic surfaces tend to resist staining by oil-soluble stains but

are more susceptible to water-soluble stains (Sears and Darby 1982). This is in agreement with the preliminary and unpublished results provided from contact angle measurements: increase in the hydrophobicity of plastic material appeared to increase the amount of oil soil residue.

The cleanability results of commercial vinyl composite tile and linoleum were in agreement with the results of Ohlson and Wäänänen (1971) and Jokelainen and Uusi-Rauva (1976a) using radiotracer technique and of Hloch et al. (1995) and Redsven et al. (2003) using colorimetry, showing that the vinyl composite tile was cleaned the best. The difference in cleanability may be due the hardness of the flooring materials. Vinyl composition tile containing quartz silica (approximately 70 wt%) is known to be harder than linoleum, which contains wood and/or cork and calcium carbonate as fillers. According to these results the described radiochemical method can be used to study quantitatively the cleanability of plastic materials with different chemical compositions in controlled study systems.

6.3 Contact angle measurement and cleanability related to surface properties

6.3.1 Contact angles and surface free energies

The drop expanding technique proved to be a feasible method to measure advancing contact angles, θ_A, on commercial flooring materials reproducibly. Increasing the drop volume in this way it was possible to ensure that the contact angle was a advancing contact angle (Kwok and Neumann 2003). The advancing drop provides a good estimation for the differences existing between the surface properties of the materials studied, especially their hydrophilicity (Michalski et al. 1998b; 1999). The measuring technique was automated to some extent, but there are several work phases from sample preparation via the placement of specimen on the measuring stage to starting the video record. However, the standard errors (SE) of advancing contact angles, θ_A, were mostly less than

±1.6˚, which can be considered sufficient for topographically and chemically heterogeneous commercial flooring materials.

The variation of θ_A might be an indication of the heterogeneity of surfaces. A liquid drop on commercial flooring materials does not follow the behaviour value predicted by the Young equation (Wenzel 1936; Cassie and Baxter 1944; Johnson and Dettre 1964), and therefore a range of contact angles was needed (Radelczuck et al. 2002; Lam et al. 2001a). The fluctuations of the contact angle values around the fitted Young equation may have been due to surface heterogeneity (Lin and Li 1995) arising from the different chemical compositions of the outermost surface layer (Meiron et al. 2004) and/or topographical heterogeneity (Wolansky and Marmur 1999). According Busscher et al. (1984), roughness below 100 nm had no influence on contact angles (Extrand 2002). On the other hand, the effect of roughness cannot be detected using this measurement system, because macroscopic angles were measured and the chemical composition of the surface layer of flooring materials probably varies.

The surface free energy (SFE) and its components were estimated using the Owens and Wendt approach. This approach has been considered to be efficient to compare various surfaces of practical materials (Michalski et al. 1998b). The calculated SFE and its polar and dispersion components are always approximations of an ideal system (Kwok and Neumann 1999, 2003; Radelczuk et al. 2002). Although the estimated values of the surface properties are tentative estimations, they are suitable for comparison of commercial flooring materials. On the other hand, there has been considerable discussion in the literature about which contact angle is meaningful, which probe liquid set should be used and which equation should be employed in the estimation of SFE and its components (Kwok et al. 1997, 2000; Della Volpe et al. 2002; Radelczuck et al. 2002). Currently there is no universally accepted method for measurement of surface free energy of solid surfaces (e.g. flooring materials). According Kwok and Neumann (1999, 2003), contact angles can be used with caution for determination of solid surface tension.

In summary, the contact angles of the probe liquids on the flooring materials were measured in a constant manner in similar conditions and the measurement technique was apparently reproducible. Thus, contact angles and the estimation of surface free energies

can be used for comparison of plastic materials and to evaluate surface properties as predictors of cleanability.

6.3.2 Cleanability related to contact angles and surface free energies

The contact angles and estimated surface free energies provided information on the relationship between cleanability and the surface properties of plastic flooring materials. Differences between the surface properties of plastic materials were sufficiently large to identify the common features of the best and poorest cleaned materials. However, water contact angle (wettability) was related to cleanability of particle- and oil-soiled flooring materials. Although the effect of surface free energy and of the polar component of SFE is tentative, these results obtained for plastic materials are promising, because they are consistent with the studies which indicated that the polar component is related to the cleaning efficiency of oil-soiled plastic surfaces (Boulangé-Petermann et al. 2003) and of particle-soiled flooring materials (Krüssmann and Garvens 1997; Redsven et al. 2003). The surface properties studied failed to explain soiling tendency. The amount of adhesion of hydrophobic oil soil has been found to be greatly dependent on solid surface hydrophobicity (Michalski et al. 1999). In conclusion, these results indicate that wettability of the material would predict the cleanability of plastic materials, a lower water contact angle indicating a better cleaning result.

6.4 Foil sampling and optical method to detect dust accumulation

6.4.1 Foil sampling and optical detection

The foil sampling and optical detection proved to be suitable to measure dust accumulation on the plastic surfaces in the offices and the effects of cleaning on these surfaces. The optically transparent gelatine foils have good sticking properties. Their sampling efficiencies have been estimated to range from 87 % to 97 % (Schneider et al. 1996). The efficiency of sampling depends basically on the extent to which particles stick to a surface, the roughness of surface, and whether the dust starts to build up in layers. Particle size dependence of sampling might follow from the fact that small particles will more easily remain in valleys of surfaces than large ones (Kildesø et al. 1999). The force (1 kp) exerted during sampling with the gelatine foil will introduce a preferred orientation. As a result, the projected area measured will be larger than the projected area of volume equivalent spheres (dust particles) (Kildesø et al. 1999). According to Schneider et al. (1996) an overall uncertainty of the sampling is caused by the positioning of foils in the detector and the natural variability due to the discrete nature of particles.

In this study conducted in the office environment the effect of cleaning was studied after one week of dust accumulation, when the surfaces were in their poorest condition. The variation of surface contamination is typically large. For this reason Schneider et al. (1994b) proposed setting a contamination level limit to the median of the individual surface concentrations. In addition, the use of the 75-percentile value as an upper limit rather than extreme values makes the results less sensitive to the occurrence of a single extreme value. On the other hand, INSTA 800 (2003) recommends the use of average values and percentiles for assessing the soiling of surfaces or controlling cleaning performance. However, to provide a representative description of the state of dust accumulation on surfaces in office buildings, the rooms are selected by using a systematic random method. In this study, the average number of samples taken before cleaning ranged from 106 to 169. According to Kildesø et al. (1995), 108 foil samples were needed

to give a reduction of 25 % in the dust-cover. In this study, the average reduction was 30% and the sample size can be considered to be representative. This method was compared by Kildesø et al. (1999) to visual evaluation. The authors concluded that visual evaluation is not as accurate as actual measurements.

To summarize, the foil sampling and optical method is suitable and easy to use in office buildings and other non-industrial environments. The sampling efficiency of gelatine foils is good. The reliability of the method can be improved by taking sufficient samples from the surfaces and by choosing the rooms of a building to be sampled with a statistical method. This agrees with Schjønning et al. (2002). In conclusion, this dust determination method is suitable in practical situations to control and develop cleaning procedures, but it could also be applicable also in scientific investigations.

6.4.2 Cleanability of office surfaces

The amount of dust on indoor surfaces is affected by cleaning methods and frequencies, by indoor and outdoor sources of soil and by the choice of surface materials. Large particles (>PM_{10}) have been found to dominate in dust build-up on surfaces (Kildesø et al. 1999). As anticipated, cleaning reduced surface contamination, except on 'surfaces close to the person' in building F. The increase of contamination in this case may have been due to the many files, papers and other objects on the tables. Thus, there was plenty of room for dust to hide or remain unremoved from the tops of files. Dust residues act as a source of resoiling if they are resuspended. Physical activities such as walking and cleaning have been found to increase the resuspension of dust particles larger than 1 μm (Thatcher and Layton 1995). According Ferro et al. (2004), the dry dusting and one person walking around contributed 32 and 15 $\mu g/m^3$, respectively to indoor $PM_{2,5}$ concentrations. In addition, differences in indoor airspeed and furnishing in a room can contribute to resuspension (Thatcher et al. 2002). Walking in the office has been found to explain from 24 to 55 % of the variation of particle concentrations in the air (Luoma and Batterman 2001). Unfortunately, the type of materials was not reported in their study. In agreement with the investigations of Kildesø et al. (1999), the greatest amount of dust was accumulated on the surfaces rated 'not easily accessible', as expected. These surfaces were

cleaned less frequently than other surfaces, according to the instructions of the cleaning programme. The lowest dust contamination was detected on the easily accessible surfaces after cleaning, which is congruent with the results of Kildesø et al. (1998). Probably the surfaces ‘close to the person’ and ‘easily accessible’ differ in their dust and soil content, e.g. some fingerprints are assumed to be present on the surfaces ‘close to the person’. In this study cleaning was performed using microfibre products, the best available method at the time. According to Nilsen et al. (2002) both dry and damp microfibre cloths remove over 90 % of the deposits from highly contaminated surfaces. Despite this the best dust quality levels of INSTA 800 (2003) were not achieved on all surfaces. However, on the well-finished and polished floor covering in building B the dust accumulation was the lowest of all the floors. This might indicate that cleaning design, including the condition of surface materials and cleaning procedures, should be taken into consideration in study buildings.

The dust contamination measurements combined with the comparison to the INSTA 800 (2003) quality guidelines for surface dust levels provides information about the current cleaning programme and the condition of surface materials in the office building studied. The information utilizes the contractors, clients, building owners and occupants in order to confirm the quality of the current situation. In office environments where a high quality level is required, cleanliness measurements are needed in order to achieve the quality requirements.

7 CONCLUSIONS

The purpose of this study was to develop and implement the methods for cleanability evaluation. The applied methods proved to be reproducible when examining the cleanability of PVC model materials and commercial plastic materials.

1. Colorimetry was a reproducible method to quantify the colour changes caused by soil in order to evaluate overall soiling and cleanability. The choice of soil type and the amount of soil appeared to be crucial for studying cleanability of materials.

2. The radiotracer method proved to be feasible to quantify soil accumulation and its removal from plastic materials. The composition of PVC model material and of soil affects cleanability during the soiling-cleaning cycles.

3. The contact angle measurement technique indicated its potential. Surface properties, especially wettability (water contact angle), appear to predict cleanability and the polar component of SFE was also tentatively related to cleanability.

4. According to the results obtained from office environments the foil sampling and optical method was suitable to detect differences in surface contamination when this sampling strategy and analysis was employed. The results can provide a tentative description of the conditions in the building and can be used for practical quality evaluation.

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