Adhesives & Sealants Industry: An Approach For Green Building Product Labelling Requirements

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SUMMARY
Adhesives, mortars, paints and all building products applied in buildings can influence the indoor air quality, both during the application and during the final users life. Emissions of volatile organic compounds (VOC) can change in terms of quality and quantity as function of building materials applied. Evaluation of VOC can be performed in different ways, according to different labels and criteria. VOC, in fact, can be considered as emitted by the product and measured in µg/m³, or as contained in the product and measured in g/l. According to ISO 16000, VOC emissions in µg/m³ have been evaluated by the test emission chamber method, built simulating a common installation of a floor. VOC, according to LEED and other international labels have been quantified in g/l .In this work differences between the methods have been enhanced, trying to correlate for one resilient flooring adhesive all data obtained following different eco labels.

KEYWORDS
VOC emissions; eco-labels; adhesives; sustainability; green-technology

1 INTRODUCTION
Adhesives for flooring systems in Europe have a long tradition for indoor air sustainability. In the 1990s, an ecological initiative tried to reduce organic solvents consumption contained in adhesives, in order to improve the indoor air quality. Solvents are considered as “liquid or mix of liquids used in manufacture of adhesives to dissolve or to dilute the binder without chemical change,” as defined in EN 923 (Adhesives-Terms and definitions, 2005.)
Solvent reduction in adhesives had both an ecological impact, with a decrease of odour and benefits on applicators health, and a safety aspect, reducing the risks of explosions and fire.
Since organic solvents have a high volatility, they have low relevance for the final consumer who is mainly exposed to long term emissions, such as terpenes, monomers and coalescing agents commonly contained in adhesive formulations.
In fact solvents considered very volatile organic compounds (very VOCs), have a high emission rate right after application that usually decreases rapidly afterwards, while semi volatile organic compounds (SVOCs) remain longer in the air. This is the reason why a solvent free adhesive should not be necessarily considered as a “low emission” one.
In order to preserve both applicators’ and final users’ health and to guarantee a very good quality in the indoor environment, some adhesive manufacturers in Europe started to develop a technology for new products which could have been considered as “low VOC emissions.”
These voluntary schemes have been approached by building products companies, starting to label the products as “very low VOC emissions” ones, where emissions are evaluated in testing chambers and results expressed in µg/m³, according to ISO 16000.
In recent years, a new ranking rule for building projects has been developed by the US Green Building Council, called Leadership in Environmental and Energy Design (LEED).

According to this standard, building materials like adhesives can contribute to one point (out of one hundred ten) in Indoor Environmental Quality credit, concerning “low emitting materials.”
An increasing number of jobsites in the US as in Europe are asking for materials complying with LEED criteria. The definition of “low emitting material” in LEED is quite different from the European definition used in all the voluntary schemes, since LEED takes into consideration the content of VOC contained in a product, expressed in g/l (Comparison of emission specifications in the US and in Europe, T. Neuhaus and R. Oppl, Indoor Air 2008)
In this work, some of the most common labels in Europe and LEED criteria for VOC evaluation have been applied on a water based adhesive, fulfilling the requirements of the different schemes.
An adhesive formulated with green technology has been chosen and tested according to Emicode, Blue Angel, AgBB scheme, and both US and Italy LEED criteria.

2 MATERIALS

A universal adhesive in water dispersion for resilient floor coverings with extended open time has been developed by R&D trying to chose the right raw materials, in order to obtain some characteristics in terms of eco-sustainability. All raw materials have been extracted in a radius of 200 km from the production plant, in order to reduce the CO_2 emissions due to the transport. Aim of the formulators is to obtain a very low VOC emissions adhesive, which can comply with all European and US green labels. This solvent free adhesive is based on a synthetic polymer, single coat in water dispersion, formulated in a ready-to-use light beige paste. The chosen adhesive is not a flammable product (flash point> 90°C), and it can be stored with no particular precautions. the formulation of the adhesive is composed by inorganic fillers (35-45%), a rosin oil (10-20%),an acrylic latex (20-40%) and water (15-30%). The acrylic latex has been qualitatively changed in the different samples analysed.

3 METHODS

Several formulations of this adhesive, with different organic latexes, have been first tested in micro-chambers (µ-CTE) in order to predict the VOC emissions and to choose the best one to be tested in the traditional chambers.
According to previous studies (Testing chemical emissions from products and materials in a routine QC environment, E. Woolfenden et al, PittCon 2010), µ-CTE can be used to predict the sample behaviour in traditional emission chambers in comparison with a reference standard. µ-CTE have a volume of ~ 45cm³, and a temperature set at 40°C; the samples are weighed and applied on an aluminium plate. Passive air sampling in this test started 24 hours after the application of 200 mg of sample in µ-CTE.
Emission chambers, made of stainless steel, have a volume of 108 l, controlled relative humidity and temperature (T= 23 ± 1°C; RH= 50 ± 3%), and loading factor 0.45 m²/m³, as requested by applied criteria. The chambers are fluxed with air, with a flow rate of 0.5 h^{-1}, which allows a complete change of the atmosphere in the chamber every two hours. The sample is mixed uniformly, weighed and applied on a glass non adsorbent surface; the test specimen is transferred into the chamber immediately after preparation. Active air sampling was collected at 1, 3, 10 and 28 days after specimen preparation. At the outlet of test chamber, exhaust air is passed through a sample tube filled with a suitable adsorbent material (e.g. Tenax TA®). Tenax tubes are then desorbed by a thermo-desorber; volatile organic compounds are separated by gas-chromatography, identified and quantified by MS detector.
As described, several criteria have been applied on the chosen adhesive: requirements and limits for different criteria are shown in the following table.

Table 1. Limits for considered criteria

Days after application	*Criteria*	*Emicode EC1^{PLUS}*	*Blue Angel RAL UZ 113*	*AgBB/DIBt*	*LEED Italy*
1 day	Carcinogenic compounds C1	-	-	-	2
	Carcinogenic compounds C2				10
	Carcinogenic compounds C3				50
3 days	TVOC µg/m³	750	1000	10000	
	Sum carcinogenic compounds µg/m³	10	10	-	
	Each carcinogenic µg/m³	-	-	10	
	Formaldehyde µg/m³	50	-	-	
	Acetaldehyde µg/m³	50	-	-	
10 days	TVOC µg/m³	-	-	-	500
28 days	TVOC µg/m³	60	100	1000	
	TSVOC µg/m³	40	50	100	
	R value	1	1	1	
	VOC without LCI	40	40	100	

LEED Italy for adhesives and sealants applies the Emicode criteria, in accordance with the edition 03.03.2009: emissions must be evaluated 1 day and 10 days after the application of the specimen into the chamber. An adhesive is considered to be LEED compliant if it can be assessed as EC1.
Concerning US LEED for schools, CA Section 01350 has been applied: products are tested from 10 to 14 days after the application of the sample on a glass substrate. Maximum allowable concentrations are limited to ½ CREL (Chronic Reference Exposure Level) for all compounds, except formaldehyde, for which the limit is set at 16.5 µg/m³ and acetaldehyde (9 µg/m³), as described in ASTM D 5116-97.
LEED US for new construction considers VOC as defined in Rule 102 (Definition of Terms, 2004) : “any volatile compound of carbon, excluding methane, carbon monoxide, carbon dioxide, carbonic acid, metallic carbides or carbonates, and exempt compounds.” VOCs are considered as the content in the can, expressed as g/l: the evaluation of VOC follows SCAQMD Rule 1168 (Adhesive and Sealant applications, 2005): a dry content (T = 110°C for 1 hour) and a Karl Fischer titration quantifying water are performed on the adhesive.
The next equation shows calculations:

$$VOC\ (g/L) = (Ws - Ww - Wes) / (Vm - Vw - Ves) \quad (1)$$

where:
Ws = weight of volatile compounds, in grams
Ww = weight of water, in grams

Wes = weight of exempt compounds, in grams
Vm =volume of material, in liters
Vw = volume of water, in liters
Ves = volume of exempt compounds, in liters

4 RESULTS

µ-CTE

Two samples of the same adhesive, formulated with different raw materials (in terms of organic compounds), have been applied on an aluminium plate and analysed in µ-CTE, at a T= 40°C. Air sampling has been performed 24 hours after the application of the products.
The next table shows results obtained as Total VOC on the two samples: values, expressed in µg/m³, are an average of three tests.

Table 2. TVOC in µ-CTE

	TVOC µg/m³	
Time h	*sample A*	*sample B*
24 h	689 *µg/m³*	169 *µg/m³*

The most abundant organic compounds detected in emissions are 2-ethyl hexanol, acetic acid and ethyl hexanol acetate.
From a first preliminary screening obtained by µ-CTE, the formulation B has been chosen for the analysis in traditional test emission chambers.

Emission chambers

Air sampling has been done 1, 3, 10 and 28 days after the application of the specimen, on Tenax tubes and on DNPH ones for aldehydes evaluation.
No carcinogenic compound has been detected at any sampling time.
Formaldehyde and acetaldehyde, evaluated according to ISO 16000-6 on DNPH tubes, are not present in the emissions after 1 day and after 3 days the application of the adhesive. Next table shows results obtained as TVOC expressed as µg/m³.

Table 3. TVOC as function of time

Time days	*TVOC µg/m³*
1 d	*1154*
3 d	370
10 d	27
28 d	5

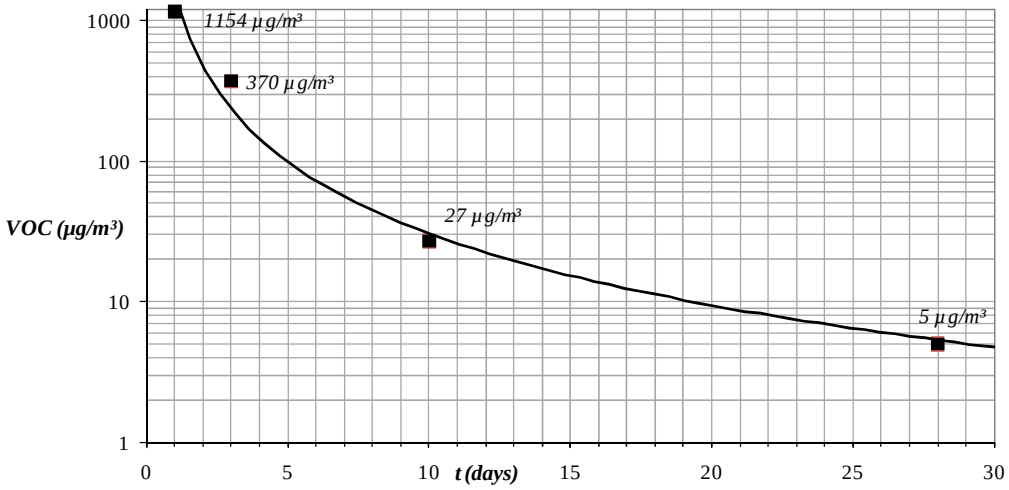

Figure 1. TVOC semi-logarithmic decay

From the TVOC point of view, the adhesive satisfies all the criteria applied.
No semi volatile organic compound has been detected after 3 and 28 days from when the test started.
Since some considered labels set the R value as a mandatory parameter to assess the adhesive as low VOC. Some substances listed in AgBB scheme are evaluated based on lowest concentration of interest (LCI) and quantified using their individual calibration factors. R value is then calculated as defined in equation (2).

$$R_i = C_i / LCI_i \quad (2)$$

The sum of all Ri, shall not exceed the value 1.

$$R = \text{sum of all } R_i = \text{sum of all ratios } (C_i / LCI_i) \leq 1 \quad (3)$$

R calculated on emissions of the considered adhesive is lower than 1.

VOC content
LEED US for new constructions, concerning adhesives, follows the SCAQMD Rule 1168 which evaluates the VOC as content in the can, expressed in g/l. The limit for this kind of adhesive is set at 60g/l.

Table 4. calculations of VOC content

Solid content %	75.0 %
Water %	24.8 %
Density g/ml	1.2 g/ml
VOC content g/l	3 g/l

5 DISCUSSION

Two water-based adhesive formulations, having the same composition but different kinds of organic fractions, have been analyzed by µ-CTE in order to hypothesize their behavior in terms of VOC emissions.
µ-CTE can be used when two or more formulations have to be compared. In this study, one adhesive analyzed with this technique at a T=40°C 24 hours after the application showed the

lowest VOC emissions: this is the reason why this sample has been chosen and tested according to the most severe European labeling schemes.
Tests performed in emission chambers confirmed that the chosen formulation could have been considered as a “very low VOC emissions” one.
Results obtained in 24 hours in µ-CTE can be used to choose the product to be tested in traditional emission chambers after a long time (28 days as asked by VOC labels).
The adhesive under consideration can be labeled as $EC1^{PLUS}$, which is the most stringent label in terms of TVOC, TSVOC and R value, so complying also with Blue Angel, AgBB scheme and Italy LEED criteria.
A parameter that can change in this criteria is the considered weight per square meter: while all the scheme in fact fixes at 300g/m² the weight of the specimen, AgBB sets the weight as the maximum signed in the technical data sheet.
In this particular study, this difference did not affect results obtained, since the two parameters are very similar.

6 CONCLUSIONS

The concept of eco-sustainability is nowadays an important issue, for building products manufacturers too. Sustainability must be recognized by the market as a fundamental value: information strategies, reference standards and investment incentives for the solutions, and barriers for not compliant products are necessary.
Benefits in indoor environment when using products with low VOC emissions are unquestionable: this is a main reason why some building product manufacturers started to study a green technology and lines of “eco” products which strongly decrease indoor pollution, consequently giving benefits to human health. This work describes the contribution of a worldwide adhesive company on sustainability and green technology, analyzing all possible formulations of an adhesive in terms of indoor air benefits.
The same approach is going to be applied to other construction products in order to certify them as “very low VOC” in markets.

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