Classification of organic substrates' wettability from contact angle measurements and hydration efficiency tests

J.-C. Michel[1,2,a], B.E. Jackson[1,b] and W.C. Fonteno[1,c]

[1]North Carolina State University, Department of Horticultural Science, 2721 Founders Drive, 27695-7609, Raleigh, NC, USA; [2]Agrocampus Ouest, UPSP EPHor, 2 rue Le Nôtre, 49045, Angers, France.

Abstract

Wettability of organic substrates was analyzed using two complementary methods in order to establish a classification based on the risk level for a material to become hydrophobic and the consequences in terms of water capture and retention. Results obtained from contact angle measurements and hydration efficiency tests were used to define 4 types of materials which have to be considered when used as substrates' components: type 1: materials always hydrophilic with no risk of hydrophobicity, types 2, 3 and 4: changes from hydrophilicity to hydrophobicity with less water retention (i.e., type 2: low and reversible risk, type 3: moderate and partially irreversible risk, type 4: high risk).

Keywords: wettability, water retention, container capacity, rewetting

INTRODUCTION

In soilless culture, the measurement of substrate wettability is very important as organic materials are used in large quantities, and many become hydrophobic during drying. Hydrophobicity can lead to a slow-down of and resistance to water infiltration and to the creation of preferential flow paths for water and solutes. This can lead to less water capture and retention of substrates and plant water uptake as well. Contact angle measurements (Michel et al., 2001) and hydration efficiency tests (Fields et al., 2014) are two different methods used for describing the changes from hydrophilicity to hydrophobicity of growing media in relation to their water content. However, the methods of analysis and their data ranges/scales are very different. The first is based on a microscale approach of particles' surface properties to develop a contact angle. The second is a more macroscale approach estimating the water capture and retention of a growing medium. The results of these two methods are not immediately transferable, because they do not measure the same things but estimate the same condition- the state of hydrophobicity in substrates.

The aim of this study was (1) to compare results obtained with both methods on different organic materials commonly used in growing media with different water contents, and then (2) to propose a classification of substrates based on their wettability. The purpose of the work is to explore the links between the two methods.

MATERIALS AND METHODS

Samples and preparation

Among the tested materials, four substrate components, representing different behaviours in terms of hydrophobicity and ability to rewet, have been chosen in this paper: wood fiber, shredded wood, fresh pine bark, and white peat (Figure 1). Wood fiber and the peat moss were provided from Klasmann-Deilmann based in Germany. Pine bark and

[a]E-mail: Jean-charles.michel@agrocampus-ouest.fr
[b]E-mail: Brian_Jackson@ncsu.edu
[c]E-mail: Bill_Fonteno@ncsu.edu

Acta Hortic. 1168. ISHS 2017. DOI 10.17660/ActaHortic.2017.1168.26
Proc. Int. Symp. on Growing Media, Composting and Substrate Analysis
Eds.: A. Baumgarten et al.

shredded wood came from North Carolina, USA; the first was sieved to have a <2 cm fraction; the second was obtained from pine logs run through a horizontal grinder, then milled to <0.6 cm.

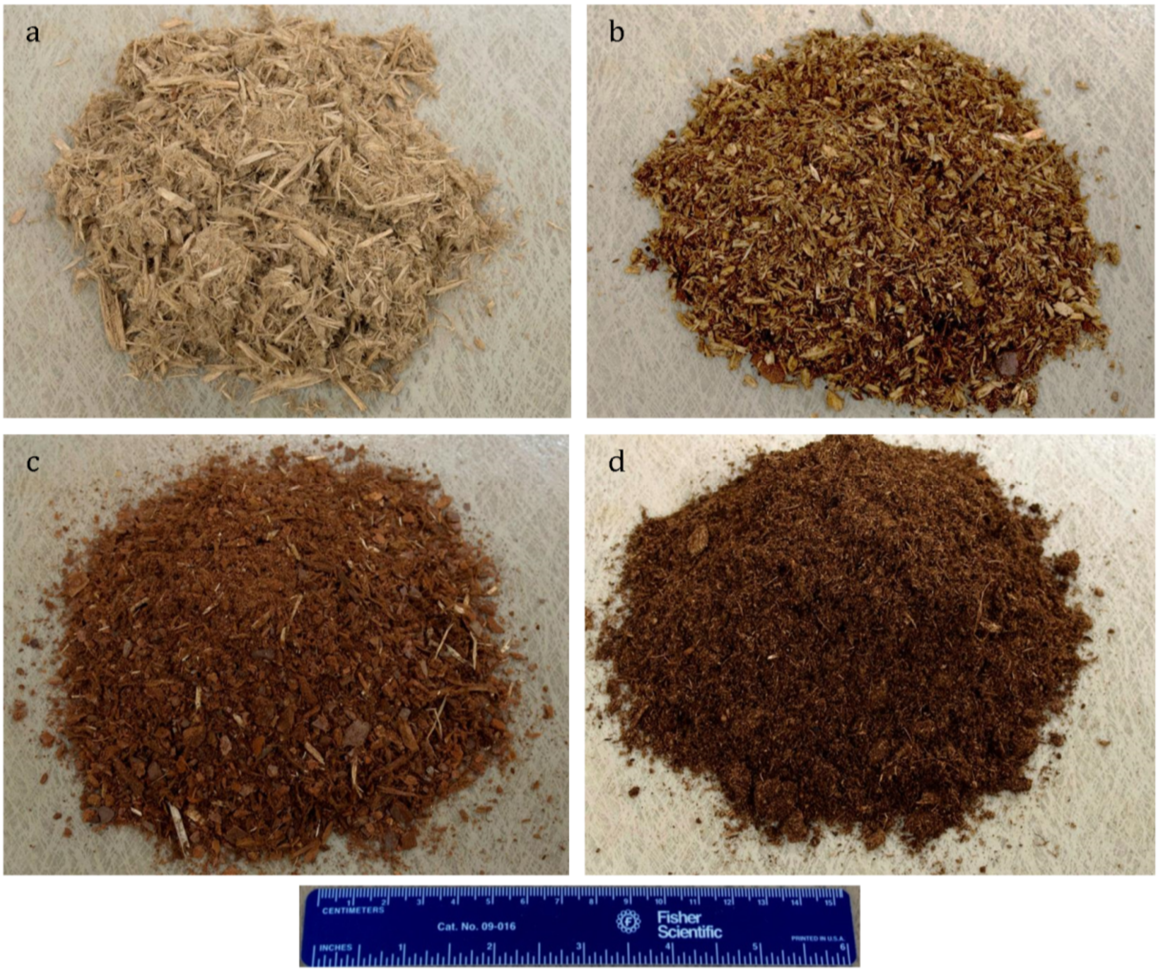

Figure 1. Materials used for these experiments: a) wood fiber, b) shredded wood, c) fresh pine bark, d) peat.

Prior to the experiments, the samples were equilibrated then maintained for (at least) one week at four initial moisture contents expressed in weight (MC): 25, 37.5, 50 and 62.5% w/w. Table 1 resumed the correspondence between initial moisture content (MC) and volumetric water content (VWC) for each material, and their dry bulk densities as well. Wettability was thus evaluated by both methods on these four substrate components, prepared at these four initial moisture contents.

Table 1. Correspondence between initial moisture contents expressed in weight (MC) and volumetric water contents (VWC) for tested substrates.

	Bulk density (g cm^{-3})				%-v/v for contact angle method and for hydration method (before 1st cycle)				%-v/v hydration method after 1st event				%-v/v hydration method after 10 events			
MC (%)	**25**	**37.5**	**50**	**62.5**	**25**	**37.5**	**50**	**62.5**	**25**	**37.5**	**50**	**62.5**	**25**	**37.5**	**50**	**62.5**
Wood fiber	0.0875				0.02	0.06	0.09	0.15	0.13	0.33	0.36	0.37	0.33	0.34	0.35	0.37
Shredded wood	0.18				0.06	0.10	0.18	0.30	0.12	0.31	0.45	0.53	0.14	0.43	0.52	0.54
Fresh pine bark	0.17				0.06	0.10	0.17	0.28	0.10	0.25	0.43	0.50	0.10	0.32	0.45	0.50
Peat	0.115				0.04	0.08	0.13	0.22	0.06	0.10	0.18	0.67	0.06	0.16	0.27	0.70

Methods for measuring wettability

1. Contact angle measurement.

Contact angles were measured by the capillary rise method described by Michel et al. (2001). This method consisted of following the capillary rise on a column of materials with water by using a Krüss Processor Tensiometer K12® linked to a computer (Figure 2). The speed of capillary rise, translated by the increase in weight of the sample, is measured in relation to time by the computer, and the contact angle was determined from the following Washburn's equation (1921):

$$\cos\theta = \frac{m^2}{t}\frac{\eta}{\rho^2\gamma_L c}$$

where θ is the contact angle (°), *m* is the mass of the adsorbed liquid (g), *t* is the time (s), η, ρ, γ_L are the viscosity (mPas), the density (g cm^{-3}) and the surface tension of the liquid (mJ m^{-2}), respectively, and *c* corresponds to an empirical constant of the porosity and tortuosity of the capillaries, which depends on particle size and degree of packing.

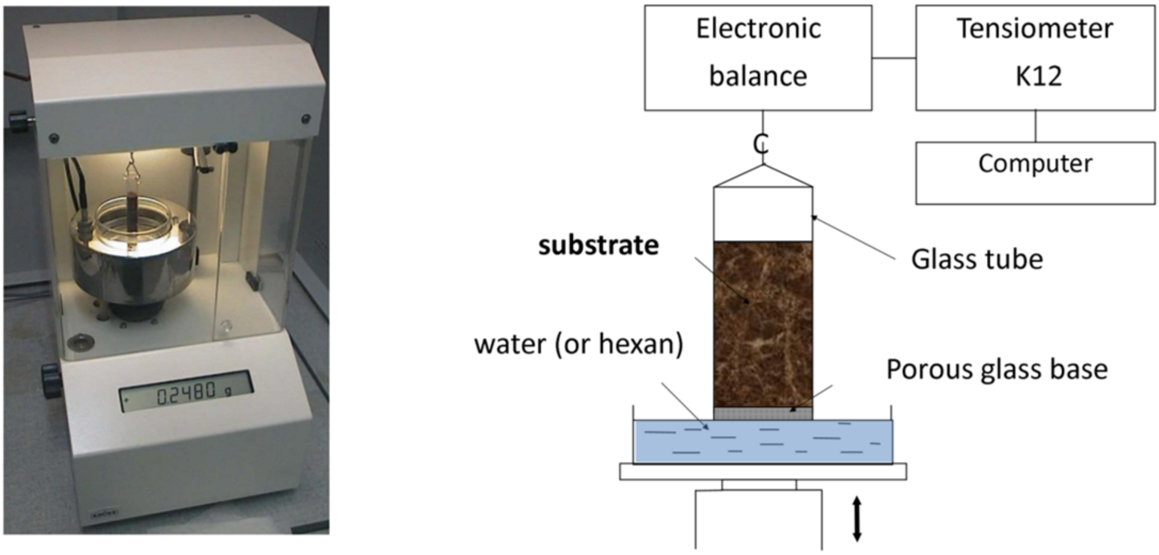

Figure 2. Instrument for determining contact angles on porous material (substrate) by capillary rise method.

The parameter c was initially assessed by using a liquid with a very low surface tension (i.e., hexane) which completely wets the sample (θ=0). The water/material contact angles could then be calculated and the wettability estimated, knowing that the greater the contact angle, the more the hydrophilicity decreases or the hydrophobicity increases. However, when the material is too hydrophobic (contact angles greater than 90°), there is no capillary rise, and the degree of hydrophobicity can then not be estimated. Contact angle measurements were carried out on a small quantity of materials (~5 cm^3) with 6 replicates $liquid^{-1}$ (hexane then water) for a given MC and for each substrate tested.

2. Hydration efficiency test.

Hydration efficiency is measured according to the method described by Fields et al. (2014). A 200 cm^3-substrate was homogeneously packed in a 10 cm height cylinder (with the objective of having the same bulk density – given in Table 1 – for a same material whatever the moisture content), then placed in the hydration efficiency unit (Figure 3a).

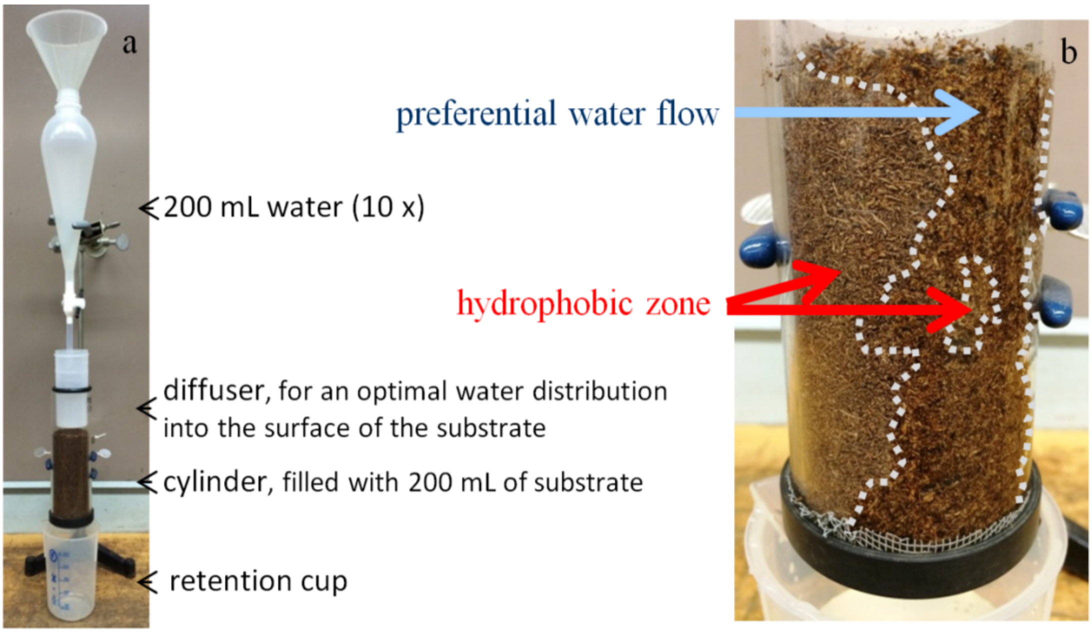

Figure 3. a) Set up of hydration device. b) Cylinder filled with peat substrate (37.5% MC), showing hydrophobic zones and water preferential flows.

The substrate column is then subjected to 10 successive hydration events which involved passing 200 mL water through each sample and to collect effluent as it came out the bottom, from which cumulative water retained in the substrate is calculated. After these 10 hydration events, the substrate column was saturated from the bottom then drained in order to determine container capacity (CC) of each material. Four replicates per moisture content and per substrate were analysed for these measurements. For the analysis and graphical representation of these results, please note that the moisture contents, expressed in weight (w/w) for the preparation of the material, were presented in volumetric water content (v/v) in Figure 4.

Correspondence between initial moisture contents (in weight) and volumetric water contents (v/v) given in Table 1 can be graphically shown for the x-axis value = 0 (i.e., Hydration Event = 0) in Figure 4. That means: 25, 37.5, 50 and 62.5% w/w represented 0.02, 0.06, 0.09 and 0.15 v/v for wood fiber; 0.06, 0.10, 0.18, and 0.30 v/v for shredded wood; 0.06, 0.10, 0.17, and 0.28 v/v for fresh pine bark; and 0.04, 0.08, 0.13 and 0.22 v/v for white peat, respectively.

RESULTS

Contact angle measurements

Before the detailed analysis of results, an important methodological limit of capillary rise has to be previously mentioned. Indeed, there is no capillary rise when a material is too hydrophobic, and hence contact angles higher than 90° cannot be measured by capillary rise method (Table 2).

As already presented by Michel (2015) in a review, contact angles increased (i.e., wettability decreased) with the intensity of drying. However, different behaviours were shown according to the tested substrates, with a general trend of decreasing wettability during desiccation from wood fiber > shredded wood > fresh pine bark > peat. In details, wood fiber remained hydrophilic in the range of moisture contents studied (25-62.5% MC), whereas shredded wood, fresh pine bark and peat showed a change from hydrophilic to hydrophobic properties with increased MC thresholds, between 37.5-25%, 50-37.5%, and 62.5-50%, respectively.

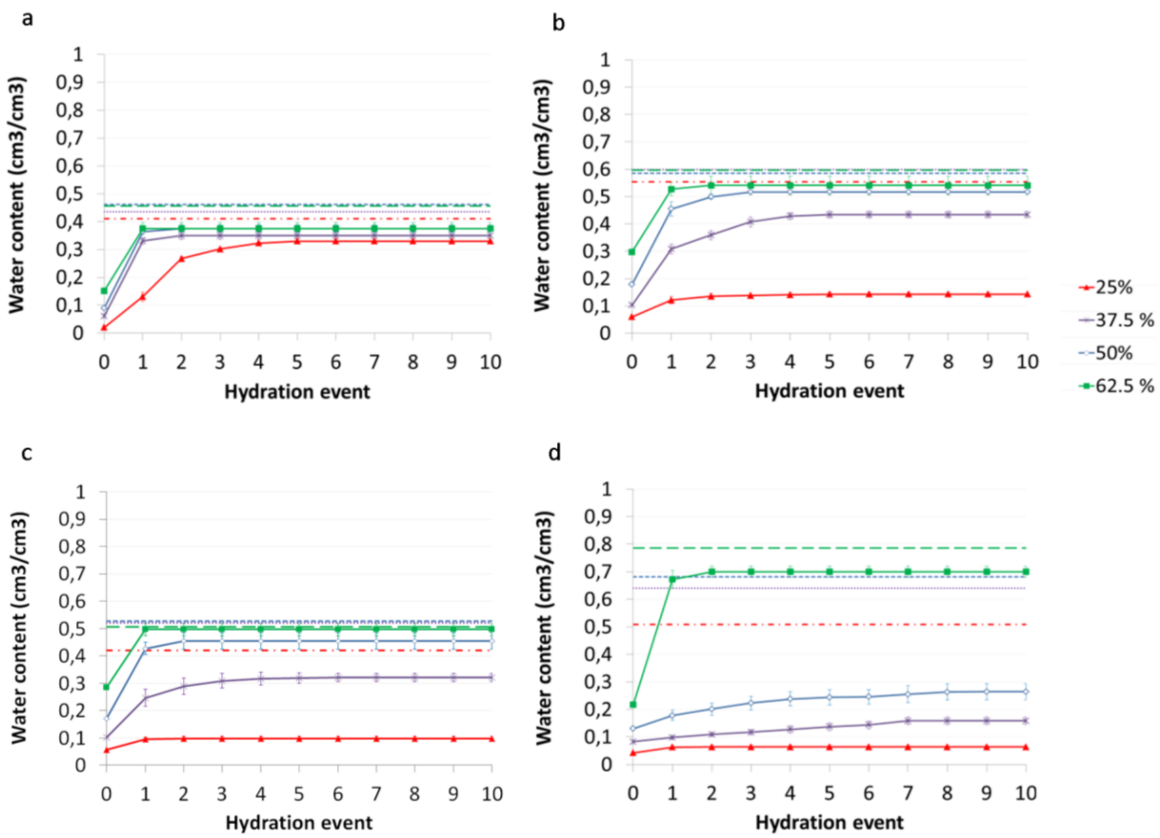

Figure 4. Hydration efficiency curves, representing the cumulated volumetric water content after each hydration event for a) wood fiber, b) shredded wood, c) fresh pine bark and d) peat. All substrate components were tested at initial moisture contents in weight of 25% (red), 37.5% (purple), 50% (blue), and 62.5% (green), translated in volumetric water content in this figure. Dotted lines represent container capacity (same set of colours).

Table 2. Contact angles measured for substrates equilibrated at 25, 37.5, 50 and 62.5% moisture contents (by weight) by capillary rise method (standard deviations did not exceed 0.3°).

Substrate	Moisture content (MC)[1] (w/w)			
	25%	37.5%	50%	62.5%
Wood fiber	87.0	82.1	81.2	77.7
Shredded wood	90.0	89.0	87.7	84.8
Fresh pine bark	90.0	89.8	87.6	82.3
Peat	90.0	90.0	89.8	85.0

[1] 25, 37.5, 50 and 62.5% w/w represented 0.02, 0.06, 0.09 and 0.15 v/v for wood fiber; 0.06, 0.10, 0.18, and 0.30 v/v for shredded wood; 0.06, 0.10, 0.17, and 0.28 v/v for fresh pine bark; and 0.04, 0.08, 0.13 and 0.22 v/v for white peat, respectively.

Hydration efficiency tests (Figure 4)

As for contact angle measurements, the behaviours during rewetting were different for the tested substrates. Whatever the intensity of drying, wood fiber quickly recovered maximal volumetric water content (~0.35) close to its CC (0.42) after only one hydration event for ≥37.5% MC and four hydration events for 25% MC. Shredded wood and fresh pine bark showed difficulties to rewet from 37.5% MC for which maximal volumetric water contents reached after ten hydration events were largely different to their respective CC (0.43 and 0.32 maximal water content vs. 0.60 and 0.51 CC for shredded wood and fresh pine bark, respectively). At 25% MC, both materials showed a strong incapacity to be rewetted, with ~0.10-0.15 maximal volumetric water contents after ten hydration events. However, CC determined for shredded wood reached a volumetric water content close to those obtained for the other initial MC (0.56 vs. 0.60) whereas differences in CC (0.41 vs. 0.51) were quite higher for fresh pine bark. That suggested a partially irreversible degradation in wettability for fresh pine bark, in comparison to the very hard but reversible rewetting for shredded wood at 25% MC. Except for 62.5% MC, the ability of peat to retain water was very low for 25, 37.5 and even for 50% MC. Moreover, CC values varied according to the intensity of drying, so that the more the substrate dried, the less its ability to rewet and retain water.

Contact angles vs hydration efficiency test (Table 3)

A first and quick observation of the relationships between the results obtained by both methods highlighted the very low ability for the substrates to capture and retain water when the contact angles were very close or equal to 90°. In contrast, substrates showed high water retention after a first rewetting event, with values in water retention close to the container capacity (CC) when contact angles were lower than 88°. Table 3 linked the minimum threshold in moisture contents, for which the changes in contact angles between hydrophilicity (θ<90°) and hydrophobicity (θ≥90°) occurred, and the ability to rewet (as described from the hydration efficiency tests) for each substrate. Results showed that the more the minimum threshold in moisture content for maintaining contact angles <90° is low, the more the ability of the substrate to capture and retain water.

Table 3. Wettability classes expressed in terms of risk level of hydrophobicity, based on their surface properties (contact angle measurements, θ) and their ability to rewet (hydration efficiency tests).

Risk level of hydrophobicity	Contact angle measurements	Hydration efficiency tests
1 No risk	θ<90°, whatever the MC	Quick recovering water contents close to CC, whatever the MC
2 Low and reversible risk	θ<90° from a minimum threshold between 25 and 37.5% MC	Recovering water contents close to CC for higher MC, but ability to rewet for lower MC quite low but reversible
3 Moderate and partially irreversible risk	θ<90° from a minimum threshold between 37.5 and 50% MC	Recovering water contents close to CC for higher initial MC, but ability to rewet for lower MC low and partially reversible
4 High risk	θ<90° from a minimum threshold between 50 and 62.5% MC	Except for higher MC, no recovering water contents close to CC. The more the substrate dried, the more its ability to rewet and its physical properties irreversibly degraded (CC = *f* (MC))

CC = container capacity; MC = initial moisture content.

θ<90° = hydrophilic substrate; θ≥90° = hydrophobic substrate.

DISCUSSION AND CONCLUSION

Despite different study-scales (micro vs. macroscopic), volumes of materials studied by each technique (5 vs. 200 cm^3), methods of watering for the substrate column (capillary rise vs. from the top), analyses of both contact angle measurements and hydration efficiency tests are very consistent and allow for establishing a classification of substrates with four classes of risk level of hydrophobicity (Table 3). These results confirmed that the ability of substrates to rewet and to recover their physical qualities is mainly controlled by changes in their surface properties (estimated from contact angle measurements). Even if this classification should be filled out using techniques allowing for the measurement of contact angles higher than 90° (i.e., droplet method), it can already be used for choosing materials as substrates' components.

Literature cited

Fields, J.S., Fonteno, W.C., and Jackson, B.E. (2014). Hydration efficiency of traditional and alternative greenhouse substrate components. HortScience *49*, 336–342.

Michel, J.C. (2015). Wettability of organic growing media used in horticulture: a review. Vadose Zone J. *14* (*6*), 0 http://dx.doi.org/10.2136/vzj2014.09.0124.

Michel, J.C., Rivière, L.M., and Bellon-Fontaine, M.N. (2001). Measurement of the wettability of organic materials in relation to water content by the capillary rise method. Eur. J. Soil Sci. *52* (*3*), 459–467 http://dx.doi.org/10.1046/j.1365-2389.2001.00392.x.