Assessment of an automated method for determining particulate organic carbon in soil

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Abstract
A manual and an automated method of sieving soil to collect a particulate organic carbon (POC) fractions were compared. The manual method is potentially subject to operator influence whereas the automated method is not. Results revealed that the amount of carbon captured in the POC fraction was larger for the automated than manual method across the five soils examined. However, the magnitude of the difference in POC as determined by the two methods varied significantly with soil type. Variation between the results obtained by different operators were also higher for the manual than automated method. The automated methodology for sieving therefore provides more consistent data across operators but also gave higher POC values.

Key Words
Soil carbon, particulate organic carbon, sieve shaker

Introduction
It is now recognised that soil organic carbon (SOC) includes a variety of different materials with different susceptibilities to biological decomposition and mineralisation. Additionally, a variety of methods have been proposed to separate and quantify the allocation of SOC to these different components based on physical and/or chemical properties. One approach uses sieving to separate a dispersed sample of soil into >50 µm particulate organic carbon (POC) and organic material associated with particles ≤50 µm (Humus) (Skjemstad *et al*. 2002). This initial sieving process is then followed by photo-oxidation and/or solid-state ^{13}C nuclear magnetic resonance spectroscopy (NMR) to quantify the amount of charcoal (resistant organic carbon – ROC) present in these fractions. Although Skjemstad *et al*. (2004) were able to demonstrate the utility of this simple fractionation scheme by successfully substituting these measureable fractions into a variant of the RothC soil carbon model, the methodology is time consuming and operator dependent. As we move towards the routine measurement of these fractions of SOC (POC, Humus and ROC) and the extension measurement and modelling across Australia, a more rapid and operator independent methodology is required.

The main operator dependence in the SOC fractionation methodology occurs during the sieving process. During this process the effort applied to cause soil material to pass through the 50 µm sieve is dependent on the operator. A new automated sieve shaker system appeared to offer the potential to precisely define and control the sieving conditions and thus reduce any operator dependence from the sieving step within the SOC fractionation methodology. The purpose of this study was to compare results obtained from the existing manual sieving approach to those obtained using the automated sieve shaker for different soils fractionated by different operators.

Methods
A series of five different soil types varying in SOC content, clay content and pH were used for this study (**Table 1**). The soils were checked from the presence of carbonate and pretreated with H_2SO_3 to remove carbonates if required (SS8 and Weisenboden soils) according to Skjemstad and Baldock (2008). Approximately 10 g of soil was dispersed in 50 ml of sodium hexametaphosphate (5g/L) by shaking for 16 hours. The dispersed suspensions of soil were then sieved according to the previous methodology or placed onto the top of a 50 µm sieve housed within an automated sieve shaking system (Fritsch Vibratory Sieve Shaker Analysette 3) equipped with the capability to spray water over the sieves as it vibrates with a defined amplitude and velocity.

In the manual system, water was passed through the sieve while a rubber policeman was used to move the soil around. It is this step which accounted for the operator dependence as the rate of water flow, rate of movement of the rubber policeman and the force applied were all subject to variation between operators. In the automated system, the amplitude and velocity of the vibratory sieve shaker and the flow rate of water through the sieves were all controlled and could not be influenced by the operator. For both systems, sieving and water flow through the sieves continued until the solution exiting the sieves became clear. The volume of water required to achieve this condition varied between soils but was typically on the order of 300-500 ml. The material retained on the 50 µm sieve (POC) was washed off the sieve into a container and dried at 60°C to constant mass and weighed. The ≤50 µm fraction (Humus) was collected in a beaker during the sieving process, transferred to a 500 ml polypropylene bottle, frozen, freeze dried and weighed. Total organic carbon contents of the POC (>50 µm material) and Humus (≤50 µm material) fractions were then determined using a LECO CR-144 carbon analyser. The POC fractions were first ground to a fine powder prior to organic carbon analysis.

To compare the manual and automated sieving methodologies, one operator analysed four replicates of all five soils using both methodologies and the acquired data was analysed as a 5 (soil type) x 2 (method of sieving) factorial design. To examine the operator dependence, three different operators analysed four replicates of three soils using both sieving methodologies and the data was analysed as a 3 (operator) x 3 (soil type) x 2 (method of sieving) factorial design. All statistical analyses were completed using Statistica 8 (Statsoft 2007).

Table 1. List of soils included in this study with some relevant soil properties

Sample	Location	Depth (cm)	Classification	pH water	C g kg^{-1}	clay (%)
SS6	Tallagalla, Qld	0-5	Dermosol	6.6	71.4	45
SS7	Toowoomba, Qld	0-5	Ferrosol	5.9	143.0	16
SS8	Waco, Qld	0-10	Vertosol	8.2	28.0	61
SS10	Gympie, Qld	0-10	Chromosol	5.0	44.4	21
Weisenboden	Adelaide, SA	0-10	Vertosol	7.7	31.0	40

Results

Comparison of sieving methods

The data acquired by one operator across all five soils and both methods of analysis are presented in **Figure 1**. ANOVA indicated the presence of a significant interaction between soil type and method of analysis. For all soils, the automated method gave higher POC contents than the manual method; however, the magnitude of the difference between the two methods varied between the soil types. SS7 had a very large difference while little difference between the two methods was evident for the Weisenboden soil. The coefficient of variation for all soils, except the Weisenboden, was lower for the automated than the manual method of sieving.

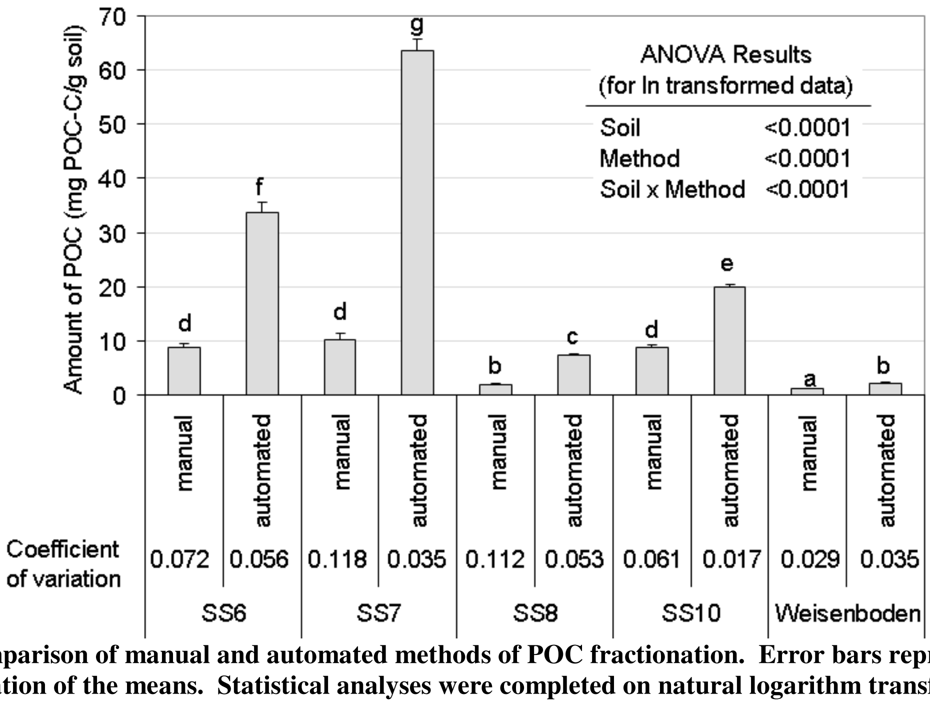

Figure 1. Comparison of manual and automated methods of POC fractionation. Error bars represent the standard deviation of the means. Statistical analyses were completed on natural logarithm transformed data to address an inhomogeneity of variance. Means with different letters had significantly different ln transformed values at p=0.05.

Comparison of results obtained by different operators analysing the same soil

The previous observation that more POC was obtained using the automated method persisted when three different operators analysed two soils using both sieving methodologies. Variation both within and between the various operators was lower for the automated method than the manual method of sieving. As a result, although the automated method gives a higher value for POC, the more consistent data was obtained across the operators using the automated method.

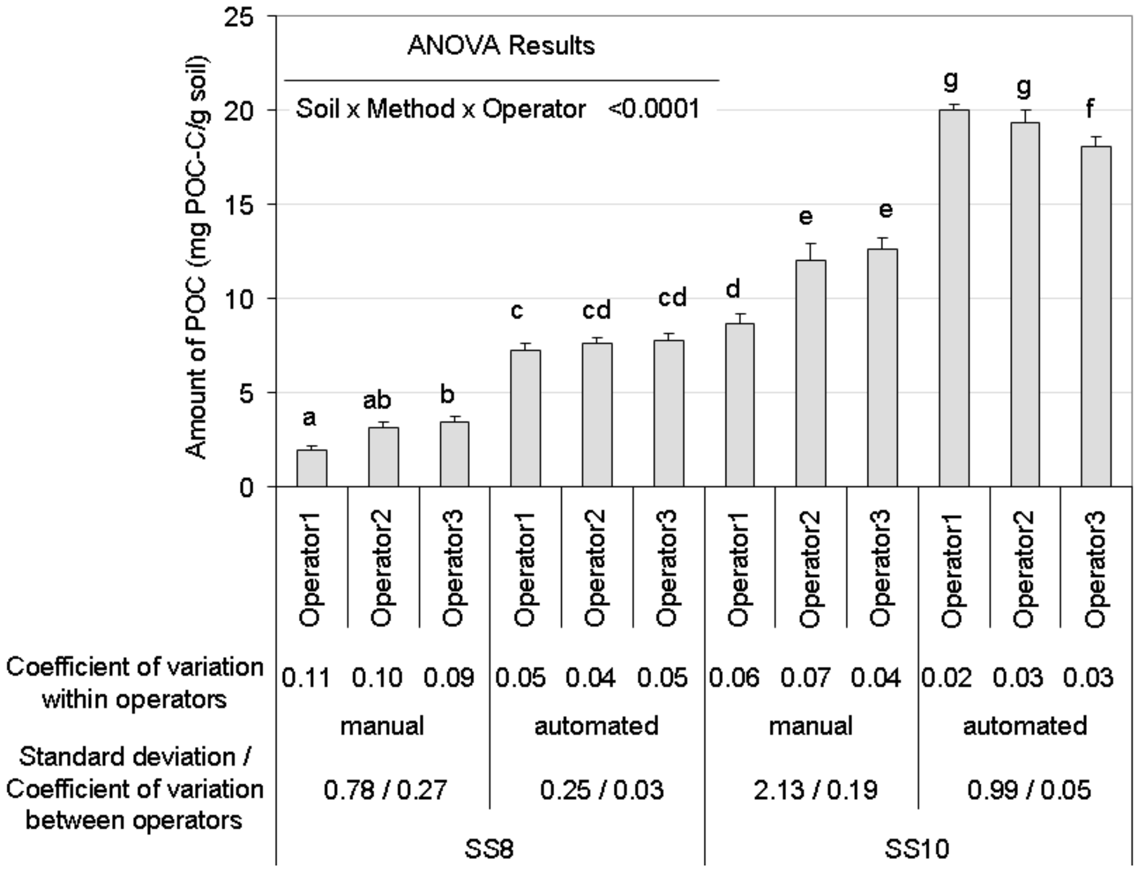

Figure 2. Comparison of operators using the manual and automated method of POC fractionation on two soils. Error bars represent the standard deviation of the four replicate determinations. All statistical analyses were performed on untransformed data. Means with different letters are significantly different at p=0.05.

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