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Chronology of plaggic deposits; palynology, radiocarbon and optically stimulated luminescence dating of the Posteles (NE-Netherlands)

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ABSTRACT

Plaggic agriculture was the dominant land use system on Pleistocene sandy soils in Northwest Europe before the introduction of chemical fertilizers. In the today's landscape plaggic deposits are still recognizable as characteristic soils and landforms, related to this historical land use system. They are also important constituents of the soil archives in Northwest Europe. In preliminary studies of plaggic anthrosols, the reconstruction of the development of the plaggic deposits was based on palynological and historical data and radiocarbon dates. Disagreement appeared between palaeopedological and historical explanation of the evolution of plaggic anthrosols. Application of OSL dating improves the knowledge of chronology of plaggic deposits. The soil organic matrix, including pollen grains, consists of a complex mix of compounds of different ages and sources. Pollen diagrams of plaggic anthrosols show a palynological registration of the Late Holocene landscape evolution, but the palynological signal is not consequently correlated with the age of the plaggic sediments. An older soil organic matrix is suspended in the voids of a younger mineral soil skeleton.

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1. Introduction

Plaggic anthrosols (ISRIC-FAO, 2006) can be considered as important constituents of the soil archives of Northwest Europe, containing high quality archaeological and palynological records (Mücher et al., 1989; Van Mourik, 1991). From the early 1940s onward the properties and age of plaggen soils have been investigated using palynological, archaeological and radiocarbon dating techniques. But accurate dating of plaggic deposits proved to be very complicated (Spek, 2004). The composition of soil organic carbon in plaggic deposits is very complex. Carbon in animal manure and litter from the standing crop is mixed with carbon from (older) humic soil horizons in the surroundings where sods were harvested (for the preparation of plaggic manure) (Fig. 1). As a consequence of these mixed constituents of different age, radiocarbon dates on plaggic deposits are unreliable. Origin uncertainty is also evident in the composition of the total pollen influx, a mix of the regular pollen precipitation and the pollen content of the plaggic manure. Dating of plaggic sediments based on palynology or radiocarbon dates of various fractions of soil organic matter never provided accurate information. Better results were expected by application of luminescence dating (Bokhorst et al., 2005; Van Mourik, 2007; Van Mourik et al., 2010). Luminescence dating techniques determine the last exposure to light of natural minerals; optically stimulated luminescence (OSL) dating of sand-sized quartz grains is the most reliable and most widely used

method (e.g. Wintle, 2008). The quartz OSL signal is reset upon light exposure and builds up due to exposure to natural background radiation after deposition and burial of the quartz grains. OSL dating is ideally suited for aeolian deposits where all grains are expected to be sufficiently exposed to daylight to have their OSL signals completely reset prior to deposition and burial. OSL dating methods are now increasingly used for establishing chronologies in other depositional environments, both natural and anthropogenic (e.g. Wintle, 2008).

Against the background of unreliable methods for dating plaggic soils, this study investigates whether optical dating methods can be applied to young, i.e. less than 1000 years old, materials, in which continued ploughing may have caused enough light exposure to reset the quartz OSL signal before burial by younger plaggic sediments.

2. Materials and methods

2.1. Sampling

A preliminary investigated plaggic anthrosols (Posteles, Mücher et al., 1989) was selected for resampling for pollen analysis, ¹⁴C and OSL dating and soil micromorphology (Fig. 2). The main reason to select this profile in the past was the nearby presence of a palynological investigated Late Holocene peat deposit (Van Der Woude, 1983). An additional reason was the fact that during the last century cultivation of the soil was restricted to the traditional ploughing technique of the upper 18 cm of the soil (oral information of the family Scholten, owner of the Posteles for at least three generations). Consequently, below 20 cm the soil archives were not seriously disturbed.

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Fig. 1. Fragment of the topographical map (1905 AC 1:25.000) with the location of the sample site. On this historical map the location Postel is indicated as Possel.

Comparison of the pollen diagrams of the peat bog and the plaggenic anthrosol resulted in a palynological based chronology of the plaggenic agriculture period. This result of this study can be considered as an excellent foundation for the evaluation of ^{14}C and OSL dating results of the plaggenic deposits of the Posteles.

2.2. Pollen analysis

Pollen extractions from humic sands were performed, using the tufa extraction method (Moore et al., 1991, p. 50). For the estimation of the pollen densities, the exotic marker grain method was applied

Fig. 2. a. Posteles in the landscape. b. profile Posteles with depth indications for OSL samples.

(Moore et al., 1991, p. 53). For the identification of pollen grains the pollen key of Moore et al. (1991) was used.

2.3. ^{14}C dating

In tradition palynological studies, radiocarbon dates were essential for the interpretation of the chronology of pollen zones in peat and limnic deposits. Also in palaeopedology, radiocarbon dates have been used for dating purposes. But it has become evident that the interpretation of radiocarbon dates of extracts of soil organic carbon is very complicated. Due to the complexity of the composition of soil organic carbon in humic soil horizons, conventional radiocarbon ages of bulk samples are not reliable. Therefore we applied radiocarbon dating on extractions of fulvic acids (FUL), humic acids (HAC) and humin (HUM) (Van Mourik et al., 1995). These fractions are based on extractability behaviour. Fulvic acids are soluble in acid and in lye, the humic acids are insoluble in acid and soluble in lye and the humin fraction is insoluble in acid and in lye. The biological decomposition rate of FUL is relatively high; they migrate easily through weak acid soil profiles or leach away completely. Therefore, they are unreliable for any dating purposes. The biological decomposition rate of HAC is medium high. Compared with FUL, they are immobile in the soil profiles and more reliable for dating purposes. The ^{14}C age of HAC is assumed to be close to the moment of fossilization. HUM (including pollen grains) will accumulate in humic topsoil during an active period of soil development; therefore, ^{14}C ages of this fraction will definitely overestimate the date of fossilized (buried) humic topsoil. It is assumed that the differences between the ages of HUM and HAC of the same level increase during an active period of soil formation. Conventional ^{14}C dating was applied on the HAC and HUM fractions of soil organic matter, extracted from samples of 70 cm (bottom of the Aan horizon) and 95 cm (2Ap horizon) depth. Samples from higher levels in the Aan horizon are contaminated by recent roots and less suitable for radiocarbon dating. For calibration of the radiocarbon dates the program wincal of the CIO (Centrum voor Isotopen Onderzoek, State University Groningen) was used.

2.4. Luminescence dating

Samples for OSL dating were collected in standard pF-rings. Under subdued orange light conditions, the samples were split: material from the outer part of the pF-rings was light exposed and was used for quantification of the environmental dose rate. This material was dried and ashed, and then ground and cast in wax. The resulting sediment-wax puck retains radon, and has a fixed geometry for measurement on a gamma-ray spectrometer which detects radionuclide concentrations (K-40 and several radionuclides in the Th and U decay chains). Radionuclide concentrations are converted into dose rates, taking into account attenuation of the dose rate due to water, organic matter, and grain size. A contribution of cosmic dose is also included. For details on the dose rate calculations we refer to Wallinga & Bos (2010).

Material from inside the pF-rings was not exposed to light during sampling, and can be used for equivalent-dose estimation. To obtain quartz extracts of grains 180–212 μm in diameter, the samples were sieved, treated with HCl and H_2O_2 and HF. The HF treatment serves to dissolve feldspar grains and etch the outer alpha-exposed layer of the quartz grains. Finally the quartz extract is washed with HCl and water, and sieved again to remove any grains that are severely affected by the chemical treatment. The samples showed negligible response to IR stimulation, indicating the absence of significant feldspar contamination. Analysis of a linearly modulated (LM-) OSL signal indicated that the OSL signal was dominated by the fast component.

Luminescence measurements used an automated Risø TL/OSL reader (DA 15) equipped with an internal Sr/Y beta source, and blue and IR diodes (Bøtter-Jensen et al., 2000). A single-aliquot

regenerative dose (SAR) procedure was used for equivalent-dose estimation (Murray and Wintle, 2003). Based on a preheat plateau test, a preheat of 225 °C for 10 s was selected, combined with a cutheat to 200 °C. At the end of each SAR cycle an elevated-temperature bleach was performed (40 s blue LED at 245 °C) to completely reset the OSL signal. Aliquots were accepted for analysis if they fulfilled the following criteria: 1) recycling ratio's within 20% from unity, 2) Recuperation values less than 10% (relative to first regenerative dose), and 3) IR signal less than 20% of OSL signal, or less than 10% depletion of OSL signal due to IR exposure. With the adopted procedure a 5 Gy laboratory dose could be accurately recovered (average dose recovery ratio 1.03 ± 0.02). To allow detection of heterogeneous bleaching only the centre 2 mm of each disc was covered with quartz grains (i.e. ~50 grains per disc). Measurements were repeated until at least 25 aliquots passed the rejection criteria. The influence of outliers on the sample mean was minimized by iterative rejection of single-aliquot estimates that were more than 2 standard deviations from the sample mean. The burial dose was then obtained from the mean of the resulting equivalent-dose distribution.

3. Results and discussion

3.1. Pollen analysis

The evolution of the plaggen agriculture in cultural landscapes on chemically poor sandy substrates is clearly registered in pollen diagrams of peat deposits (Van Der Woude, 1983; Van Mourik, 1987; Van Mourik and Pet, 2001). Important features in pollen diagrams are the Late Holocene decline of arboreal pollen and the rise of *Calluna* and *Cerealia*, related to the anthropogenic deforestation and the extension of plaggen agriculture. Additional palynological markers are the appearance of *Fagopyrum* around 1350 AC and extension after 1450 AC (Leenders, 1987) and the extension of *Pinus*, due to Pine plantations on the former *Calluna* heath after 1900 AC, when sod digging and plaggic deposition stopped after the introduction of chemical fertilizers (replacing the plaggic manure).

Mücher et al. (1989) showed that pollen analysis of the Posteles profile revealed a record of successive vegetation and cultivated plants, comparable with the results of pollen analysis of an adjacent peat deposit in a vanished brooklet at about 600 m distance of Postel (Van Der Woude, 1983). They also pointed to a remarkable synchronism of the development of cereal prices in NW-Europe (Bieleman, 1987) and the fluctuations in the *Cerealia* curve in the pollen diagrams of the peat deposit and the plaggic anthrosol.

However, the composition of pollen spectra is complicated due to the various sources of the pollen grains: pollen produced by the in situ vegetation, pollen dispersed by the regional vegetation and pollen present in plaggic manure. Still, just as in peat bogs, aspects of environmental development are clearly reflected in pollen diagrams of plaggic anthrosols.

Fig. 3 shows the pollen diagram of the resampled profile Posteles.

The pollen content of the 2Ap and 2B horizon post-sedimentary infiltrated by bioturbation and agriculture in sterile deposited Late-Glacial coversand. Characteristic is the sharp decrease of pollen concentrations with depth, shown by the pollen density curve. Pollen grains could survive in the well preserving micro environment of (welded) excremental aggregates, mainly produced by micro arthropods (Figs. 4, 5) (Van Mourik, 1991). The spectra of the 2B horizon reflect already evidence of agriculture (*Cerealia*) in a deforested landscape (low percentages of *Alnus*, *Quercus*, *Fagus*). The spectra of the 2Ap horizon show increasing percentages of *Cerealia*. The combination of high percentages *Cerealia* pollen and the abundant presence of intertextic distributed welded organic aggregates in the soil matrix (Fig. 6) points to crop production, using organic fertilizers without any mineral component.

The pollen content of the Aan horizon (plaggic deposits) is sin-sedimentary. The soil matrix (Fig. 7) consists of mineral grains, mainly

Fig. 3. Pollen diagram Posteles.

quartz grains. Welded excremental organic aggregates are present in the voids of the mineral matrix but also visible in a cutanic position. The pollen spectra reflect the extension of *Fagopyrum* and the oscillations of the curve of *Cerealia* and *Alnus*, preliminary observed by Mùcher et al. (1989). The sods for the preparation of plagic manure were partly collected on heath lands (*Ericaceae*), partly on wet grasslands (*Cyperaceae*).

The present Ap horizon (the active plough horizon) is characterized by the highest percentages of *Cerealia*, a slight extension of *Pinus* (planted on the abandoned heath after 1900 AC) and the rise of *Zea Mais* (introduced in Dutch agriculture after 1950 AC).

3.2. Radiocarbon dating

The radiocarbon ages of the HAC fractions are younger than the ages of HUM fractions of the samples from 70 and 95 cm depth (Table 1).

Fig. 4. Intern fabric of an aged welded organic aggregate (fine organic skeleton grains, organic plasma, charcoal particles). The absence of fine double fringing mineral particles is indicative for a micro environment where organic decomposition is controlled by fungi and micro arthropods.

Compared with the palynological age, the radiocarbon dates clearly overestimate the age of plagic deposits. This is the consequence of the heterogeneous composition of organic matter, in situ decomposed by micro arthropods into excremental aggregates. The input is a mixture of collected humic sods, containing earlier produced excremental organic matter, 'fresh' animal manure and 'fresh' litter from cultivated plants. The intern fabric of the excremental aggregates consists of a dense packed mixture of cellulose and lignin fragments, charcoal particles, fungal remains, pollen grains and organic plasma. Because of that it is unreliable to correlate the radiocarbon dates of plagic deposits with their sedimentary ages.

3.3. OSL dating

Previous research has shown that reliable OSL dating on decadal to centennial timescales is possible where light exposure prior to deposition is sufficient to reset the OSL signal in all grains (e.g. Ballarini

Fig. 5. Pollen grain in aged welded organic aggregate; the acid and micro-environment of excremental aggregates explains the pollen preservation in well drained humic soil horizons (Van Mourik, 2001).

Fig. 6. Soil skeleton of the 2Ap horizon, showing an intertextic distribution of (abundant) welded organic aggregates.

et al., 2003; Madsen and Murray, 2009). In fluvial settings, where bleaching conditions are less favorable, advanced statistical methods are needed to obtain reasonable age estimates (e.g. Wallinga et al., 2010). Equivalent-dose distributions provide a good indication whether light exposure prior to deposition and burial was sufficient to reset the OSL signal in the majority of grains, provided that measurements are made on small aliquots or single grains (e.g. Wallinga, 2002a,b; Duller, 2008). For this study we made use of such small aliquots.

It is unlikely that all quartz grains deposited in the plaggic soils are exposed to sufficient light to completely reset the OSL signal prior to deposition. We expect part of the grains to be of aeolian origin, whereas another part will be introduced with sods and manure. After deposition there are additional bleaching opportunities. Ploughing will mix the top layer of sediments on the plaggic field, resulting in repeated light exposure of the grains during the period that they are inside the plough horizon. Spek (2004) assumes during the application of manure from sods a mean ploughing depth between 15 and 20 cm and a mean plaggic sedimentation rate of 15–20 cm per century. Based on these figures, quartz grains, added to the soil in plaggic manure, can be maximal part of the furrow for 100 year. Grains are permanently shielded from daylight after burial below the plough horizon; from this point onward the OSL signal will build up. Hence we expect the OSL age to determine the time of burial of the grains below the plough horizon.

Equivalent-dose distributions for all but the lower two samples of our study indicate tight, symmetrical distributions with few outliers. The equivalent-dose distributions are shown in Fig. 8). This suggests

Fig. 7. Soil skeleton of the Aan horizon, showing a partly intertextic, partly cutanic distribution of welded organic aggregates.

Table 1

Chronology of the plaggic deposits of profile Posteles.

Depth	Horizon	OSL age	Palynological age	¹⁴ C age HUM	¹⁴ C age HAC
0 cm	Ap	–	1900 AC	–	–
20 cm	Ap	–	1760 AC	–	–
45 cm	Aan	1758 ± 14 AC	1650 AC	–	–
59 cm	Aan	1711 ± 20 AC	1550 AC	–	–
70 cm	Aan	1651 ± 31 AC	1450 AC	1071–257 AC	1159–1253 AC
82 cm	Aan	1626 ± 20 AC	1350 AC	–	–
95 cm	2Ap	1517 ± 31 AC	–	789–890 AC	893–1025 AC
105 cm	2Ap	2035 ± 450 BC	–	–	–

that light exposure prior to burial beneath the plough horizon was sufficient to erase the OSL signal in the vast majority of grains, a result that is in line with preliminary measurements reported on plaggic deposits near Weert (southern Netherlands) by Bokhorst et al. (2005). We expect the ages on these samples (ranging from 1626 ± 20 AD to 1758 ± 14 AD, all in stratigraphic order) to provide accurate estimates of the time of burial below the plough horizon.

The lower two samples (top and bottom of the 2Ap horizon) show more scatter in their equivalent-dose distribution. The sample from a depth of 95 cm shows a distribution that is slightly wider than those of the shallower samples. Also, there are more outliers at the higher end of the distribution, indicating that light exposure was less favorable for resetting of the OSL signal in all grains. As a consequence, we cannot exclude the possibility that the OSL age of this sample (1517 ± 31 AD) slightly overestimates the true burial age. The lowermost sample shows a much wider distribution, with all aliquots returning doses greater than the average equivalent dose determined for each of the overlying samples. This may reflect partial resetting of the OSL signal of part of the grains due to limited light exposure, but may also be caused by mixing of sand grains of two layers of different age (the top of the coversand deposit and the bottom of plaggic deposits) without light exposure. Probably the bottom part of the 2Ap was hardly affected by bleached mineral grains in plaggic manure deposits. During traditional cultivation before the start of plaggic deposition, only part of the grains were light exposed since coversand deposition in the Late Glacial.

4. Evaluation of the results for the chronology of plaggic anthrosols

Table 1 summarizes the results of OSL, palynological and ¹⁴C dating. The differences are evident, but what is the significance for the evolution of the plaggic anthrosol?

4.1. Radiocarbon ages (Table 1)

Soil micromorphological observation show the complex composition of soil organic matter. The welded aggregates of the 2Ap and the Aan horizons show a complex composition of organic skeleton fragments and plasma and even incorporated pollen grains and charcoal fragments and the sources of all these compounds can differ in ages. The pollen spectrum of the sample of 95 cm depth shows low percentages Arboreal pollen and the appearance of Cerealia. The radiocarbon dates (800–1000 AC) are maybe an indication for the transition from a forested to an agricultural landscape. The radiocarbon dates of the sample of 70 cm depth seems to overestimate the age of the introduction of the land use system, applying plaggic manure. Pollen grains are always part of the HUM fraction of soil organic matter together with lignin, cellulose and charcoal particles. Some of these particles are the result of in situ decomposition processes, other particles are inherited from sods and even aeolian deposits. The result is an overestimating of the radiocarbon age of the extracted soil organic matter and its pollen content.

Fig. 8. The equivalent dose distributions of the six samples of Posteles.

4.2. Palynological ages (Table 1)

Mücher et al. (1989) proved the correlation between the pollen diagrams from the Posteles and the adjacent peat bog and the history of cereal prices around the North sea basin. Based on these correlations, they decided to the palynological based chronology of the Posteles deposits (table 1). Considering the development of soil organic matter in fertilized agricultural soil systems, it is clear that radiocarbon dates overestimate the 'historical' ages of pollen spectra. Based on the palynological data, plaggic deposition started after 1350 AC and stopped around 1900 AC. Mücher et al. (1989) calculated for a deposition period of 450 year a mean annual plaggic deposition rate of 1.60 mm/year.

4.3. OSL ages (Table 1)

The OSL ages are correlated with the moment of burying of quartz grains under the active plough horizon. That means that OSL ages are

closely correlated with the mineral ages of plaggic deposits, not with palynological or radiocarbon ages. Based on OSL ages, the plaggic accumulation started around 1600 AC. That indicates a mean sedimentation rate of 3.0 mm/year. Note that in the active soil the bleaching of quartz grains continues by ploughing; at the same time there is a continuous accumulation of organic matter, including pollen grains. That explains at least a part of the differences in palynological and OSL ages of the same samples of plaggic deposits (Fig. 9).

5. Conclusion

OSL and ^{14}C and ages of the plaggic deposits differ significantly. OSL dates correlate with the age of the mineral skeleton. Radiocarbon dates do not correlate with the age of the deposits, due to the mixture of organic compounds of various sources. Radiocarbon dates overestimate the age of plaggic deposits. Pollen grains are incorporated and preserved in soil organic aggregates and consequently part of the soil organic matrix. Dating of plaggic deposits based on historical and

Fig. 9. OSL and palynological ages of the plagic deposits of profile Posteles (the use of plagic manure continued till 1900 AC, the year of the introduction of chemical fertilizers).

palynological data may overestimate the age of plagic deposits slightly.

Information from pollen diagrams of plagic anthrosols is relevant for the reconstruction of the evolution of cultural landscapes on chemical poor sandy substrates. But the ^{14}C and palynological ages of these paleo-ecological records are not significant for the correct dating of the plagic deposits. The application of OSL dating proves that an older soil organic matrix is suspended in the voids of a younger mineral soil skeleton.

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