Chemical analysis of residues from seventeenth-century clay pipes from Stratford-upon-Avon and environs

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ORGANIC RESIDUES ASSOCIATED WITH 17th-century clay pipe bowls and pipe stems from England, including samples from the site of William Shakespeare's residence in Stratford-upon-Avon, have been chemically analysed. Compounds firmly identified from this pilot study include nicotine, myristic acid (known to be hallucinogenic), borneol and other forms of camphor. Also identified were vanillin, quinoline and cocaine introduced to Europe from South America. Residues of *Cannabis* are suggested but not proven. It is not assumed that any of the pipes were necessarily used by Shakespeare, but the results support the view that at least one hallucinogen was accessible to him and other writers in the 17th century.

Background

It has been suggested that William Shakespeare was at least aware of the hallucinogenic properties of one or more substances, including perhaps *Cannabis sativa,* a plant which was certainly accessible in Elizabethan England for paper, rope, garments and sails.[1] One possible way to determine whether hallucinogenic substances were smoked in Shakespeare's time is to undertake chemical analysis of clay pipes of the kind that have been discovered at the site of New Place in Stratford-upon-Avon, Shakespeare's residence in the early 17th century. Clay pipes from this site were reported by Law.[2] Additional pipe fragments from the Stratford area have subsequently been discovered. Although research has been undertaken on the size, shape and style of pipe bowls to estimate the ages of such artefacts,[3,4] apparently no chemical analyses have been undertaken to determine what substances other than tobacco may have been smoked in England during the 17th century.

It has been generally assumed that 'tobacco pipes' were used only for the smoking of *Nicotiana* products introduced to England by Sir Walter Raleigh and others who returned from exploits in the New World. Even in Shakespeare's time, however, 'wild tobacco' or 'Indian tobacco' could refer to *Cannabis.* A recent interpretation of texts that include Shakespeare's reference to a 'noted weed' suggested the possible use of *Cannabis* as a substance that affects the mind.[1] This article serves as an initial report of a pilot study of 17th-century clay pipes from England, to determine what substances in addition to *Nicotiana* ('Virginian' tobacco) might be identifiable from organic residues associated with fragmentary pipe bowls or stems.

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Materials and methods

Twenty-four pipe fragments were made available by the Shakespeare Birthplace Trust in Stratford-upon-Avon. Some of this material has contextual information, e.g., 'New Place' (site of the residence of Shakespeare and his son-in-law, Dr John Hall, who would have had access to imported compounds for medicinal purposes;[5] 'Harvard House', which was occupied by the mother of John Harvard; 'Shakespeare Birthplace garden'; Loxley, situated just outside Stratford-upon-Avon; and Abingdon, Oxfordshire. Specimens in this study include several from the Cuthbert Coves-Jones collection curated by the Shakespeare Birthplace Trust.

Internal bowl diameters of English pipes from the 17th century are generally less than 12 mm.[3,6] Measurable specimens used in this study include bowls with a mean internal diameter of 10.6 ± 0.8 mm (n = 9). On the basis of style, shape and size, all of the pipe bowl samples in this study probably date to the 17th century.

Most of the pipe fragments examined have been cleaned by museum curators, but in several instances black or grey substances adhere to inner walls of the bowls and probably include products of combustion from the time the pipes were used. A few pipe bowls and stems retained soil associated with black flecks, and these were included for analysis.

Residues and sediments from the interior of bowls and bores of pipe stems were treated in 5 ml chloroform to extract organic compounds. The organics were then concentrated in 0.2 ml solvent and analysed by coupled gas chromatography-mass spectrometry (GC-MS) using an HP 5890 gas chromatograph interfaced with a 5890 mass selective detector. The gas chromatograph was equipped with a 30-m, 0.25-mm internal diameter, J&W DB-5 capillary column. Helium was used as the carrier gas. The injector temperature was kept at 250°C and that of the transfer line at 280°C. The oven temperature was programmed as follows: 50°C initial temperature for 1 minute, increased to 110°C at a rate of 30°C per minute and kept at 110°C for 2 minutes, then increased to 280°C at a rate of 17°C per minute and kept at 280°C for 10 minutes.

Identifications were based on EI spectra expressed in terms of mass:charge ratios (m/z) and relative abundances of compounds in reference samples (Wiley electronic library).

Substances such as *Cannabis* degrade within short periods and can be difficult to identify after combustion in clay pipes. Traces of compounds derived from *Cannabis* may, however, be identified,[7–9] including cannabidiol and cannabinol, which are produced by heating.[7] African clay pipes from archaeological contexts more than 600 years old have been previously shown to have *Cannabis* residues.[10] Mass:charge ratios of 193, 231, 238, 243, 246, 258, 271, 295, 299, 310 and 314 are indicative of compounds derived from *Cannabis,* though the effects of heating and age may influence positive identification. Cannabinol and cannabidiol are associated with m/z values of 310 and 314, respectively.[7]

Results

Among the substances positively identified in this study (Table 1) were nicotine, myristic acid and isopropyl myristate, cocaine, cinnamaldehyde, vanillin, quinolene and butyl quinone, borneol and other forms of camphor, pyrene, phenol, toluene and naphthalene. All of these substances can be identified with a high probability relative to reference specimens listed in Table 2. Compounds associated with m/z values of 193, 231, 238, 243, 246, 258, 271, 295, 299, 310 and/or 314 have been found in samples 1912.6, 1912.8, WS-3, WS 5A, WS-7C, WS-8, WS-9 and WS-10, but intensities associated with these measurements were low.

Discussion

Nicotine is expected for clay pipes that became popular for smoking tobacco in

Table 1. Organic substances identified from a sample of 24 pipe fragments available from the Shakespeare Birthplace Trust, Stratford-upon-Avon, including fragments from the Cuthbert Cove-Jones collection (CCJ). WS-3, WS-8, WS-9 and WS-10 come from the site of New Place, residence of Shakespeare and his son-in-law, Dr John Hall; WS-1,WS-2, WS-5A, WS-5B, WS-5C, WS-6A and WS-6B are from the Shakespeare Birthplace Garden; WS-7A, WS-7B and WS-7C from Harvard House, Stratford-upon-Avon. Internal pipe bowl diameter (BD). Residues (R) adhering to pipe fragments, or sedimentary infill (S) from bowls and stems, were analysed using gas chromatography linked to a mass spectrometer.

Sample	Context	Pipe	R/S	BD (mm)	Substances
1912.1	CCJ	Bowl	R	10.6	Pyrene
1912.2	CCJ	Bowl	R	11.0	Nicotine
1912.3	Stratford	Bowl	R	9.5	Camphor
1912.4	CCJ	Bowl	R	10.6	Camphor, phenol
1912.5	CCJ	Bowl	R	10.3	Camphor
1912.6	Abingdon	Bowl	R	9.8	Cocaine, camphor, toluene, phenol, *Cannabis*?
1912.7	Loxley	Bowl	R	–	–
1912.8	CCJ	Bowl	R	11.0	Borneol, toluene, naphthalene, *Cannabis*?
1912.9	Loxley	Bowl	R	10.3	Toluene
1912.10	CCJ	Bowl	R	12.5	Camphor
1939	Stratford	Bowl	R	–	–
WS-1	Stratford	Bowl	S	–	–
WS-2	Stratford	Bowl	S	–	–
WS-3	Stratford	Bowl	R	–	Cinnamaldehyde, vanillin, butylquinone, *Cannabis*?
WS-4	Unknown	Bowl	S	–	Naphthalene
WS-5A	Stratford	Stem	R	–	Phenol, naphthalene, *Cannabis*?
WS-5B	Stratford	Stem	R	–	Isopropyl myristate
WS-5C	Stratford	Stem	R	–	–
WS-7A	Stratford	Stem	R	–	Cocaine
WS-7B	Stratford	Stem	R	–	–
WS-7C	Stratford	Stem	R	–	Myristic acid, *Cannabis*?
WS-8	Stratford	Stem	R/S	–	Carotene, phenol, *Cannabis*?
WS-9	Stratford	Bowl	S	–	Quinoline, *Cannabis*?
WS-10	Stratford	Bowl	S	–	*Cannabis*?

17th-century England. Borneol is known to occur naturally in *Dryobalanops aromatica* in Borneo, while other forms of camphor are known from China. Samuel Purchas,[11] a contemporary of Shakespeare, described Borneo camphor as 'the best'. Camphor may have been used in 17th-century England to offset the unpleasant smell associated with odours of tobacco smoke, which King James I recognized as unhealthy.[12]

Myristic acid is known to occur in Myristicaceae, a family of plants that includes nutmeg, the seeds of which produce oil which has been studied chemically[13] and recorded as having hallucinogenic properties.[14] Nutmeg was a commodity highly prized by European explorers including Ralph Fitch, who returned to England in 1591 after travelling to the East Indies in search of spices. An entry in Fitch's journal has been associated with a line in Shakespeare's *Macbeth* (Act 1, Scene 3): 'Her husband's to Aleppo gone, Master of the *Tiger*', identified with a journal entry[15] in which Fitch writes 'I did ship myself in a ship at London, called the *Tiger,* wherein we took the way to Aleppo'.

Cinnamaldehyde, or 'Cassia aldehyde', is derived from plants of the genus *Cassia,* including *C. fistula* from India, which was used for medicinal purposes but we note that it has also been referred to as a 'pipe tree'.[16]

Unequivocal evidence for *Cannabis* has not been obtained. This may be attributable to difficulties associated with the effects of heating, and problems in identifying traces of cannabinoids in old samples. Specimens WS-7C, WS-9, and 1912.6 all have substances associated with *m/z* values of 314, potentially indicative of cannabidiol, which is a product of heating *Cannabis.*[7] These specimens and samples WS-3, WS-5A, WS-8, WS-10 and 1912.8 registered *m/z* values of 193, 231, 238, 243, 258, 295, 299 and/or 310, but the intensities of the readings were too low to provide certain identifications. The results are suggestive but do not prove the presence of *Cannabis.*

Toluene may be derived from more than one source, including balsam. In *Timon of Athens,* Shakespeare refers to balsam in the context of its use in medical practice. The presence of toluene in clay pipes was unexpected, but it may be a product of the smoking of substances such as *Cannabis.* Toluene was found in pipe 1912.6, which registered low intensity readings at *m/z* values 193, 231, 295, 299 and 314, and pipe 1912.8 indicated toluene together with low intensity readings at 193 and 310 (*cf.* cannabinol), suggestive of *Cannabis* that has been heated and is present in very small quantities.

Quinoline and butylquinone, from samples WS-3 and WS-9 respectively, are possibly derived from the South American *Cinchona* plant, introduced from Peru to Europe by Spanish Jesuits in the seventeenth century. Quinine, derived from the powdered bark of the same plant,[17] was used for the treatment of individuals suffering from malaria, including a Spanish countess in 1638. The presence of quinoline in 17th-century pipe residues suggests the use of the South American *Cinchona* plant in a way not previously recorded historically.

Cocaine from South American *Erythroxylon* is an unexpected result. It was associated with pipe stem WS-7A from Stratford-upon-Avon (unwashed specimen from Harvard House), and pipe bowl 1912.6 from Abingdon, with internal bowl diameter (9.8 mm) and style consistent with a 17th-century date. The material analysed from Stratford sample WS-7A came from the narrow bore of a pipestem that had not been exposed to possible contamination (such as the handling of modern paper money with traces of cocaine).

Table 2. Mass:charge ratios (*m/z*) associated with substances in samples listed in Table 1 and compared with reference samples used for purposes of identification, using gas chromatography-mass spectrometry.

Substance	Mass:charge ratios	Reference number
Nicotine	51,65,84,92,105,119,133,161,162	47308
Borneol	41,51,55,67,71,91,95,110,121,136,139	40594
Camphor	55,68,81,95,108,123,137,152	37916
Phenol	57,67,73,91,105,105,115,121,145,155,161,177,205,220	105772
Toluene	41,57,67,81,91,105,115,121,145,161,177,189,205,220	106135
Pyrene	43,57,71,101,150,174,202	87361
Cocaine	42,82,94,105,122,166,182,198,272,303	180589,180591
Cinnamaldehyde	51,77,103,131	22091
Vanillin	81,109,123,137,151	37226
Quinoline	51,63,89,101,106,115,128,140,143	30484
Butyl quinone	41,67,91,107,121,135,149,163,177,192,220	105434
Isopropyl myristate	43,55,60,67,73,83,97,102	153673
Myristic acid	43,55,60,73,83,87,97,102,115,129,143,157,166,171,185,199,228	114506
Carotene	41,69,95,121,137,273,299,341,367	264886
Cannabis	193,231,238,243,246,258,271,295,299,310,314	
Naphthalene	51,75,160,195,230	115547

Cocaine was introduced from South America to Europe in the 16th century, initially through Spanish conquistadors[17] who in turn were raided by English explorers such as Sir Francis Drake, a contemporary of Shakespeare.

In Sonnet 27, Shakespeare refers to 'a journey in my head', potentially associated with a 'trip' and 'out of body travel' in an altered state of consciousness.[1] In Sonnet 38, he refers to a 'Tenth Muse' (as a source of inspiration?). In Sonnet 76 he refers to 'invention' (*cf.* the act of writing) in the context of a 'noted weed', and questions why he should turn away from 'new found methods' and 'compounds strange'. The lines in this sonnet can relate to 'new literary styles and unusual combinations',[18] but may also refer to chemical substances. In other Shakespearean contexts (*Cymbeline* 1,v.8), 'compounds' refers to 'compounded drugs'.[16,19]

'Compounds' is an interesting term that deserves to be explored further in literary as well as chemical analyses. We suggest that, in a play on words, Shakespeare was using the word 'compounds' to refer not only to new combinations of words such as those used by Chapman, but also to new substances that were being introduced to England in the 16th and 17th centuries at a time of exploration and discoveries of new organic substances.

One of Shakespeare's contemporaries, Alexander Craig, wrote sonnets that express 'wild changes in mood',[18] including reference to a 'Pype of Loame', that is, a clay pipe:

I carouse each day, from Pype of Loame and for thy saike

I souke the flegm-attractive far-fett Indian smoke.

In this context, 'Indian' could relate to the smoking of Indian hemp, 'wild tobacco', 'Indian tobacco' or *Cannabis sativa,* which has anti-phlegmatic properties[20]; or alternatively, 'Indian' in the verse could relate to substances from American 'Indian' territories.

Not all of the compounds identified from the pipes were necessarily smoked. Carotene in one sample (WS-8) from New Place garden may be derived from organic material associated with soil in which pipes were embedded. Naphthalene in three samples (1912.8, WS-4 and WS-5A) is possibly associated with former curatorial practice to control insects in museum collections with mothballs.

Some of the substances identified in this study may be contaminants, but the identification of nicotine is consistent with the generally accepted view that pipes of the kind we examined were used primarily if not entirely for the smoking of tobacco. Spices such at nutmeg were used for medicinal purposes, as attested by Dr John Hall's records,[5,21] but the curious combination of substances that were known to have been introduced to England at the time of the spice race suggests that at least some people were experimenting with a variety of newly imported products in the context of smoking.

We do not assume that any of the bowls and stems were from pipes used by Shakespeare. However, this study supports the suggestion that at least one hallucinogen was accessible in England in the 17th century, and may have been used by writers.[1]

The notorious association between narcotics and creativity has been assumed to be a post-Romantic phenomenon in European culture, but our research gives reason to question this assumption.

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First gene sequence of a flowering plant

Among the latest milestones in the elucidation of the genetic make-up of living organisms is the first complete sequencing of the genome of a flowering plant, announced last December. The species concerned is thale cress (*Arabidopsis thaliana*), which is related to broccoli and cauliflower, though it is not a food plant.

A multi-national team took 10 years to discover that *Arabidopsis thaliana* has approximately 26 000 genes, distributed among 5 chromosomes, comprising some 117 million pairs of nucleotide bases. Approximately 3000 genes have been studied so far, some 800 of which appear to be vital for photosynthesis. Most *A. thaliana* genes have counterparts in the fruit fly *Drosophila melanogaster* and the nematode *Caenorhabditis elegans,* which implies common ancestry. The plan now is to work out within 10 years what every gene does.

There are more than 250 000 species of flowering plants, on which we depend for food and oxygen. By discovering everything about the genetic blueprint of thale cress, researchers hope to be in a good position to improve the disease resistance and productivity of food plants generally, for example, and perhaps even understand better how human genes function.

A suite of articles on the *A. thaliana* genome work was published in the 14 December issue of *Nature* (vol. 408**,** pp. 791–826).

As we go to press, we learn that two U.S.-based companies, Syngenta and Myriad Genetics, have announced their sequencing of '99.5%' of the rice genome — the cereal crop that helps to feed half the world's population.

This gives the companies a three-year lead over a publicly funded attempt to disclose the 430 million nucleotide bases of the genetic map of rice. Jubilation over this great achievement is tempered by concern that access to information about the genome, which is desirable for anyone wishing to improve rice yields or pest resistance, will come at a price dictated by the companies. Genetic information from the publicly funded research, when complete, would be freely available.