

ABSTRACT

Two ceramic smoking-pipe bowls, excavated in Lalibela Cave, Begemeder Province, Ethiopia, were radiocarbon dated to 1320 ± 80 A.D. A modified thin-layer chromatographic technique, applied to the pipe residues, yields positive tests for cannabis-derived compounds. Long-lived cannabinoids, produced by the heat of smoking from short-lived psychoactive ingredients in cannabis, makes identification possible.

The origins and spread of *Cannabis sativa* are obscure, although the plant has achieved worldwide distribution, and is commonly known under such names as bhang, ganja, dagga, hemp, marijuana, etc. The psychoactive properties especially of its tropical varieties are well-known as they have apparently been since the 3rd millennium B.C. The plant is mentioned in a 2737 B.C. pharmacological treatise attributed to the Chinese emperor Shen-Nung (Merlin 1972). Another possible reference occurs in Vedic texts from India around 2000-1400 B.C., while Herodotus gives a clear description of the Scythian practice of throwing hemp seeds on hot rocks in a confined space. By 950 A.D. the use of cannabis was well-established in Arabia; Marco Polo's account of the alleged hashish-related assassin's cult of Hasan-ibn-al-Sabbah (11th century) is also well-known, if not necessarily accurate. We now have chemical evidence that the plant was smoked in Ethiopia in the 13th-14th century A.D.

According to Vavilov's phytogeographic postulates, the site of species formation of *Cannabis sativa* was in central or Southeast Asia; the plant was probably domesticated in the same region. The cultigen is of the same species, while a more strongly psychoactive form has been named *Cannabis indica*; the latter may or may not be a separate species.' Since the same psychoactive compounds are involved in both cases, the differences can be ignored here. The main psychoactive constituent is tetrahydrocannabinol (THC); this, along with cannabinal and cannabidiol, is the substance commonly tested for in the chemical identification of cannabis (Turk et al. 1969). They occur naturally only in the cannabis plant (THC has been produced synthetically). All three compounds degrade at least partially when burned (as in smoking), and also deteriorate rapidly with time. These attributes provide difficulties when testing for evidence of cannabis use in residues from ancient smoking pipes.

Two ceramic pipe bowls, excavated by J. C. Dombrowski (1971) at the site of Lalibela Cave in the Begemeder Province of Ethiopia (near Lake Tana), were tested for the presence of cannabinolic compounds. The pipes came from level 2 of the cave, with an associated

radiocarbon date of 1320 ± 80 A.D. (Y-2433); calibration on the bristlecone pine curve produces no significant change in age. This date is clearly earlier than the introduction of tobacco to Africa from the New World, following Columbus' journey. Archaeological remains from Lalibela level 2 differ but little from the present-day material culture of the region, and the workmen at the site were able to identify the pipe bowls and their mechanical operation. Both bowls formed part of waterpipes; an aperture at the bottom of the bowl allows for the attachment of a vertical stem, which presumably descended into a water container. Pipe B2 was well preserved, contained a "pipe-cake" as thick as 1 mm in places, and was identified as an Awraja's pipe or the pipe of a "big man." Pipe A2 was broken and contained only a small amount of residue.

A standard thin-layer chromatographic technique, used in hospital and police testing for the presence of cannabis, was used in the experiment.² A small sample (less than 100 mg) of the unknown (plant material or pipe residue) is soaked in petroleum ether, with appropriate stirring to break up caked materials. (Ether can also be poured directly through a pipe if no obvious residue is present.) Cannabinolic compounds dissolve in about 10 minutes. The ether solution is then poured off, evaporated in a stream of hot air, and the residue is reconstituted with a few drops of chloroform. Using a capillary tube, a drop of the solution is "spotted" about 3 cm from the lower edge of a 20 x 20 cm glass plate, coated with a 250 micron layer of silica gel G. Spots from different samples are spaced about 2 cm apart, the same distance from the edge. The plate is then placed upright in a container with about 1.5 cm of benzene in the bottom, the container tightly covered, and left for 30 minutes. During this time the benzene rises about 15 cm in the silica gel. As it passes the sample spots, cannabinolic compounds separate and travel upwards on the plate for a distance proportional to their affinity for benzene. The distance travelled, relative to the total height reached by the benzene, provides the R_f value of a given compound. When the plate is removed from the benzene container and sprayed with a solution of fast blue B salt (naphthanil diazo dye, K & K laboratories), cannabinolic compounds appear as orange-red spots. In testing fresh samples of *Cannabis sativa*, a major spot (THC) developed at an R_f value of 0.53, with lesser spots at 0.62 (cannabinol) and 0.45 (cannabidiol).

Residue samples from the two Ethiopian pipes (A2 and B2) were compared on the same plate with a 60% THC resin extract and positively identified samples of *Cannabis sativa*. The standards produced the customary spots in each case, while A2 and B2 did not. Two faint spots, representing unidentified cannabinoids, developed from A2 and B2 at R_f values of 0.69 and 0.84; these spots were absent in the standards. In a second experiment, a 60% THC resin standard and several modern pipes which had previously tested positively for cannabinolic compounds were used for comparative purposes. The THC standard and the modern pipes exhibited the familiar spot for THC, cannabinol, and cannabidiol, while pipes A2 and B2 did not; the modern pipes exhibited faint spots at R_f values of 0.69 and 0.84, as did pipe B2 (from which sufficient residue had been available). In a third experiment, no spots could be developed from A2 and B2 (probably due to insufficient sample material), but modern pipe samples once again developed faint spots at 0.69 and 0.84. Lack of sample material prevented further testing of A2 and B2.

A third pipe, excavated by J. Atherton from a pre-tobacco context in Liberia, yielded no results in three attempts. The pipe lacked visible residue, and ether was poured through it in an attempt to obtain sample material.

We conclude that THC, cannabinal, and cannabidiol have relatively short lifetimes and cannot be identified in ancient samples. (A figure of 50 years is often mentioned, but remains untested.) When subjected to the heat of smoking, however, one or more of these compounds degrade to form two unidentified cannabinoids, with Rf values of 0.69 and 0.84, and lifetimes in excess of 500 years. The presence of these cannabinoids in a pipe positively identifies its use for the smoking of Cannabis sativa or one of its relatives or extracts. The evidence is difficult to detect, with success probably dependent on sample size (not determined) and on the strength of the material smoked (highly variable).

Archaeologically, we conclude that some variety of Cannabis saliva was smoked around Lake Tana in the 13th-14th century, in much the same way as it is today. This is evident for pipe 132 (two positive tests), somewhat less so for A2 (one positive test). How and when the plant, and knowledge of its psychoactive properties, reached this area is unknown; an Arabic source seems probable.³ In view of these results it may be necessary to review archaeological interpretations relating to smoking pipes in early African contexts. A prime example could be the chronological system based on pipe designs which has been established in West Africa, where the exclusive use of tobacco has been assumed.

REFERENCES

DOMBROWSKI, J. C.

1971 "Excavations in Ethiopia: Lalibela and Natchabiet Caves, Begemeder Province." Boston: Boston University Ph. D. Thesis.

MERLIN, M. D.

1972 Man and marijuana. Teaneck, New Jersey: Fairleigh Dickinson University Press.

TURK, R. F., et al.

1969 Method for extraction and chromatographic isolation, purification and identification of tetrahydrocannabinol and other compounds from marihuana. Journal of Forensic Sciences

14:385-391.

1 Editor's note: see paper by Schultes in this volume.

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3 Editor's note: see paper by du Toit in this volume.