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Hot or not: systematic review and laboratory evaluation of the hot needle test for microplastic identification

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Abstract

Microplastics are small (< 5 mm) synthetic polymers that are a contaminant of emerging concern and can be difficult to identify due to their diversity in size, shape and composition. The hot needle test, or hot point test, helps researchers identify suspected microplastics under optical microscopy by probing their physical melt or deformation behavior, and is a low-cost and practical method for widespread use. However, to our knowledge the accuracy of this test has not been fully evaluated. We noted that articles commonly referenced by researchers for the hot needle method do not have a detailed description nor evaluation of the method accuracy. To address this knowledge gap, we took a mixed methods approach to describe the conditions under which the hot point test performs accurately, including a systematic literature review, reporting of the response of known fibers to a hot point, and evaluation of method performance by researchers in both controlled and environmental samples. In a single-blind trial of researchers applying different hot point conditions to a set of synthetic, semi-synthetic and natural fibers, synthetic and some natural fibers were correctly identified > 70% of the time. While cotton and semi-synthetic fiber results were less consistently identified (< 65% correct), this was improved (82–100% correct) in a second trial when clearer, updated guidance was given regarding the difference between a "pass" and "fail" response, showing the potential for the hot needle test to help analysts avoid false positives. Cellulose acetate from cigarette filters was the most challenging to identify because although this material may melt, response of individual fibers to heat varies and can be difficult to observe for smaller microfibers. Reported confirmation rates by spectroscopy of suspected microplastics that pass the hot needle test vary widely in the literature. Using detailed hot needle test criteria, > 90% of microplastics that we selected from environmental samples (water, sediment) were confirmed by Raman microscopy. It is recommended that researchers assess their hot needle test methods against known standards of both target microplastics and background materials like natural fibers, report the response criteria used in their studies and optimally include spectroscopic verification of results for higher confidence.

Keywords Microplastic, Hot needle test, Hot point test, Melt test, Method evaluation

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Introduction

Plastic is a ubiquitous material in the built and natural environment and the levels of this pollution, which has resulted from the exponential rise in the production, use and disposal of plastics during the twentieth century, poses a threat to the earth system [23, 58]. Defining the levels and potential impacts of plastic debris from macro to micro to nano in diverse environments has also grown and taken considerable effort in method development among researchers (e.g. [47, 61, 73]. Microplastics, defined as synthetic polymer particles in the size range from 1 micron to 5 mm, are a diverse contaminant class, consisting of different types (e.g. fibers, fragments, foams, spheres, films) and compositions (polymers, additives) and have received a lot of attention since their relatively small size makes them available for long-range transport and exposure to a wide range of organisms [64].

There are several approaches that researchers take to identify suspected microplastics. Most commonly, after isolation from a matrix, suspected microplastics are counted under magnification by an optical microscope using visual identification clues [47, 61, 64]. The accuracy of visual identification alone has been shown to be variable and quite low in some cases, especially for small microplastics (i.e. < 100 micron), with possibility for microplastic enumeration to be either under- or over-reported [43, 68]. A confirmation technique is therefore needed for quality assurance. It has been advised that some fraction of suspected microplastics (e.g. > 10%) should be analyzed using spectroscopic methods, such as FT-IR and Raman spectroscopy, which are non-destructive and can identify polymer composition [30, 35]. Hyperspectral imaging for microplastic analysis is an emerging field [22]. Microplastics in samples may also be dyed during sample processing (e.g. Nile Red technique) and identified by fluorescence microscopy, although issues with specificity of this technique are raised since other types of organic matter may absorb dyes and therefore care must be taken to avoid over-estimation [28, 50]. Mass spectrometry has also been applied to identify microplastic composition, although sample preparation and analysis is destructive [73]. Given instrument specifications and method detection limitations, different approaches are perhaps best suited depending on targeted microplastic types, size ranges or matrices. However, these methods are variably costly, time-intensive and require the use of specific instrumentation that many labs cannot access. Therefore, another approach is to combine visual identification under optical microscopy with response of individual particles to physical probing, although this is also labor and time-intensive and presents a lower size limit on the particles targeted [47, 61]. Physical probing includes response to touch (e.g. elasticity vs breakage) and heat (e.g. melt behavior via a "hot needle test" or "hot point test").

The "hot needle test" (or "hot point" test, to encompass tools other than needles) is a method used in many studies but the technique is not standardized and description is often vague [2, 34]. The basic principle is that if the suspected plastic reacts to the hot point, it can be counted as a positive identification. A defined positive response is limited to verification of polymers that react in a specific manner, with melting rather than charring or decomposition occurring (i.e. most easily observed for thermoplastics and thermoplastic elastomers). A given polymer type will have a range in temperature over which it softens or melts, depending on various factors including molecular weight, crystallinity and composition (e.g. copolymers, polymer blends, plasticizers and other additives). Another issue is that the specific visual behaviors may be subjective. The possibility of false identifications could lead to over- or under-estimation of plastic counts in samples.

The objectives of this work are to determine if the hot needle/hot point test (hereafter referred to by the more common phrasing, hot needle test (HNT)) is effective at identifying suspected microplastics. To accomplish this, we i) synthesize use, description and validation of the method in the literature by a systematic review, ii) classify the reactions of some known fibers to exposure to a hot point to help in method guidance, iii) assess variation in method performance among researchers by conducting single-blind trials of the method under different conditions, and iv) test method accuracy by Raman microspectroscopy on two sets of unknown suspected microplastics isolated from environmental samples that either pass or fail the hot needle test.

Methods

Systematic literature review

A systematic literature review was conducted to find research articles published through June 2022 using the hot needle test to verify suspected microplastics. We queried the database Web of Science, which searches title, abstract and key words, for the term "microplastic*", excluding topic areas not relevant to environmental science and excluding proceedings, data papers and retracted publications and found 7624 articles. The top 20 journals publishing these articles are shown in Table S1. Based on these results, we chose to query Science Direct (the discovery platform for Elsevier journals), as well as Environmental Science and Pollution Research (Springer journal publishing), Nature Scientific Reports, Royal Society of Chemistry and American Chemical Society journals on the journal websites. Through these platforms we approximate having at least ~ 60% coverage

of the microplastic literature based on the Web of Science query results. Web of Science was not used for the literature review because it does not search full text, and articles usually do not have "hot needle" in the title, abstract or keywords.

We queried the literature databases listed above for research articles with the terms "hot needle" and "microplastic*" or "hot point" and "microplastic*". While we found 12 hits using the term "hot point", several were review articles or were research articles that also used the term "hot needle" elsewhere, and thus only 4 research articles were identified using only the term "hot point" in the full text. We sometimes encountered alongside the term "hot needle test" in the literature the term "melt test", but this is not a common name for the method. Review papers (37) were removed from search results. Since the focus was on the use of this method in research articles, papers (23) mentioning the use of a "hot needle" as part of a literature review, and not methods, were also excluded. Overall, 131 research articles (see all references in Supporting Information) were identified as using the hot needle test in some way in their method to probe suspected microplastics in diverse matrixes (atmospheric deposition, water, sediment, sewage sludge, and biota).

Known fiber response to hot metal point (soldering iron)

A range of synthetic, semi-synthetic and natural fibers were tested using the hot needle test and a melting point analyzer. Fibers were selected for this study because they are often the most common type of microplastics encountered in samples as well as difficult to identify visually. Fibers were prepared from sewing threads or garments, Spartina marsh grass (dead and living) was collected from local marshes, and weathered cigarette butts of various brands ($N=8$) were collected during a roadside litter sweep. Textiles threads included polypropylene (IG Design), polyester-coated cotton (65%/35%, Coats and Clark), cotton (Gutermann), nylon (6,6; pantyhose), acrylic (polyacrylonitrile; sweater), polyester (polyethylene terephthalate; Gutermann thread and fleece robe), cellulose acetate (cigarette filter), viscose (Gutermann), and silk (Gutermann). Polymer identity of garments found on labels and of cigarette butt filters was confirmed by FTIR-ATR (Bruker Alpha). All fiber materials were tweezed apart down to as close to a singular fiber as possible. Given that behavior could change due to chemical alteration during sample processing in microplastic isolation methods, we chose two cellulosic materials, spartina grass and cellulose acetate cigarette filters, to also test as either digested or undigested. The digestion consisted of submerging the material in a 1 M KOH solution for 72 h, a common protocol for isolating microplastics from organism tissue.

Several single threads of each material were taped on one end to a glass slide and probed with a temperature-adjustable soldering iron (Hakko FX-888D) equipped with extra fine tips set to 350 °C while recording with a digital microscope camera (Opti-TekScope, OT-HD). Response to heat with a short contact time (~ 1-2 s) was described (e.g. melt, movement or significant shrinking/deformation or change in shape). An additional measure was taken to determine the response of a cotton fiber to a heated point by ramping the temperature of the soldering iron in 50 ℃ intervals from 150 ℃ to 350 ℃.

The melting points/behavior for the materials were researched in the literature and several were also measured using a digital melting point analyzer (Mel-temp Digital Melting Point Apparatus). This information was used to check that the hot point (e.g. soldering iron) temperature setting was hot enough to cause an appropriate reaction from the synthetic fibers and to provide insight into hot needle test responses.

Single-blind hot needle test trials and survey

A total of 10 local microplastics researchers were recruited for a survey and single-blind microfiber identification test to evaluate the hot needle test (HNT) method in two trials (HNT 1 and HNT 2). In both trials, a unique Petri dish for each participant was set up with 9 different fiber types cut to lengths < 300 micron and randomized on a square grid. Two or three fibers of the same type were placed in each grid as a test of replicability. The fibers tested in HNT 1 included polyester, acrylic, nylon, cotton, silk, viscose, undigested spartina grass, undigested cellulose acetate and digested cellulose acetate; in HNT 2 they were the same except undigested spartina was changed to polypropylene. All fibers were in the same neutral color palette (near white/cream/clear), so as not to have bright colors potentially bias the test. In both trials, participants were recruited from laboratories using the hot needle test as part of their protocols who had been trained in microplastic identification and comfortable with optical microscopy techniques. In HNT 1, participants ($N=8$) conducted the test in one of two laboratories where they typically worked and were allowed to use whatever method of HNT preferred. Each participant recorded their technique in the accompanying survey data sheet (see Supplemental information). Four of the eight participants in HNT 1 were recruited for HNT 2. In HNT 2, participants ($N=6$) performed the test at the same location, were given specific instructions to use a soldering iron set to 275 °C and the criteria for a positive response. The participants filled out the data sheet counting the number of fibers in each grid, stating their color and how many passed or didn't pass the HNT, and their accuracy was then evaluated using a key. Numerical

values were given for the fraction of fibers in each grid that were correctly identified by participants. For example, if a grid cell contained 3 polyester fibers and the participant recorded that 2 of the 3 passed the hot needle test, then a score of 0.66 correct was given. Descriptive statistics on the scores were calculated by individual and by material. We tested whether the proportion of correctly or incorrectly identified fibers was significantly different from random guessing (50:50) in both HNT trials using a Chi-square goodness of fit test (alpha = 0.5, DF = 1).

Raman spectroscopy analysis of hot probe-tested environmental samples

Raman microspectroscopy was used as a secondary verification analysis to assess the performance of a hot needle test using a soldering iron set to 250 °C or 275 °C in two different sample sets. Suspected microplastic fibers isolated from pond water that passed ($N = 8$; representing 10% of total) as well as several that did not pass ($N = 7$) according to criteria developed for HNT 2 (see Supplemental Information and Table 1) were analyzed. All were > 63 micron and of various colors (yellow, black, red, blue). In brief, grab samples ($N = 4$) were collected from two stormwater ponds in coastal South Carolina. The samples were filtered onto a 63-micron stainless steel sieve and the retained contents were rinsed into a clean glass jar using filtered DI water, into which hydrogen peroxide was added to make a 3% H_2O_2 solution and samples digested for at least 72 h to reduce organic material. The second set of samples included suspected microplastic films, fragments and fibers ($N = 63$, ~ 12% of total counts) isolated from street dust and urban sediment. In brief, sediments were dried in an oven at 60 °C, sieved to 63–500 micron, density separated with saturated NaCl (1.2 g/cm^3), digested with 20% H_2O_2 solution at 40 °C on a hot plate for 1 week then sieved > 63 micron to remove fine particulates. For both sample sets, contents after digestion were vacuum filtered onto cellulose nitrate gridded filters. Soldering iron temperature was set to 250–275 °C because the cellulose nitrate filter reacts strongly to a hotter probe. Selected particles were transferred from the filter using metal tweezers to double-sided tape affixed on an aluminum dish, encircled and numbered using a fine-tipped permanent marker and logged with notes about color/category and analyzed by micro-Raman (Horiba-Yvon XploRA Plus Raman microscope; 785 nm laser excitation, 1 s acquisition time sampling binned up to 200 acquisitions, 600 gr/mm grating, 10-100X magnification, slit 100 micron, aperture 300 micron, spectral range 400–2000 cm^{-1}). Material identification was aided by an on-site polymer library as well as the SLoPP and SLoPP-E Raman [54] and Open Specy [15] microplastic spectral libraries with match quality > 0.7.

Table 1 Criteria for performing the hot needle test (provided to participants in HNT 2)

Fiber "passes" when it either: melts (i.e. softens, changes size, turns from solid to liquid) or bends/curls (distorting near point of heat contact)
Fiber "fails" when there is: no response, charring/burning, or wavering (moves slightly or recoils but doesn't significantly change shape or size)

Results and discussion

Systematic literature review

Across the papers using the hot needle test that were reviewed ($N = 131$; full list provided in Supplemental information), about 40 different studies are cited for the hot needle test method; however, the most cited are De Witte et al. 2014 [17] (32%) and/or Devriese et al. 2015 [18] (20%). The top 9 studies cited for the hot needle test method are shown in Table 2, and collectively are cited by 82% of the papers reviewed. A citation for the method was not provided by 15% of papers reviewed. An additional 32 studies were cited at least once and they were reviewed for any additional method information provided, but most of them cited other papers in their methods and a few were incorrect citations (i.e. no mention of the hot needle test was in the cited publication). De Witte et al. 2014 [17] is the most commonly cited study for the hot needle test likely because it was one of the first to gain attention as using it, and then other researchers cited it as a method origin. The description of the hot needle test in [17] is "Each plastic fragment was verified as plastic with a hot needle." Devriese et al. 2015 [18] state only "The hot point will make the plastic sticky and leave a mark". Neither publication provides the type of needle used nor how it was heated.

The most common descriptions in literature research methods are that microplastics when pressed or a hot needle comes near will react or respond by melting, curling, deforming, or becoming sticky or adhering to the needle. Papers citing Devriese et al. 2015 [18] commonly reaffirm the description of the hot needle making the plastic sticky with a dark mark [10, 11, 39, 55, 63]. Others state that the microfiber will move or contract [9, 16, 37, 60], which may be a similar observation as to "curl". However, "move" is a vague descriptor to interpret, as it could also refer to a simple movement without a structural change of the material. Rather than a needle, Helmberger et al. 2020 [28] used a soldering iron with a specified operating temperature of 390℃. Norén [56] doesn't describe using a needle, but rather heating particles on a glass slide (Table 2). Very few papers indicate what type of needle (material, hollow/solid) is used, or how they are heated. A stainless-steel hypodermic syringe needle [4,

Table 2 Publications most cited for the hot needle test method in the present systematic review

Reference	# citations (% of papers reviewed)	Description (and references where given)
De Witte et al. 2014 [17]	42 (32%)	"Each plastic fragment was verified as plastic with a hot needle."
Devriese et al. 2015 [18]	26 (20%)	"The hot point will make the plastic sticky and leave a mark."
Campbell et al. 2017 [14]	8 (6%)	"application of a heated needle tip to each plastic to confirm that it would melt. Although characterization using Raman spectroscopy would have been helpful in identifying the nature of the plastics, budgets did not allow this."
Lusher et al. 2017 [46][a]	8 (6%)	"Perhaps the simplest technique is the use of a hot needle to observe melting points [6, 17, 18, 70]. While both cheap and fast, this method does not allow for the accurate identification of the polymer; however, the temperature range at which melting occurs does provide a specific range of potential plastics."[d]
Karlsson et al. 2017 [36]	5 (4%)	"On occasion the hot needle test, where a hot needle is pushed against the particle to test if/how the material melts, was also used."
Barrows et al. 2017 [3][b]	5 (4%)	"The Hot Needle Test (based on [17]: In the presence of a very hot needle, plastic pieces will melt or curl…When using this technique, be sure your needle is very hot and held as close as possible to the piece in question (without blocking your view). If the needle is not hot enough, you will see no movement, even if the piece is plastic. This test should be used in conjunction with knowledge of other characteristics of plastic pieces."
Silva et al. 2018 [67][a]	5 (4%)	"the use of a heated needle tip to each plastic particle to ascertain whether the suspected particles melt when subject to heat. Nonetheless, this method has the drawback of not allowing for the identification of the polymer in question, although it remains a viable approach, particularly when more expensive equipments, such as spectroscopic analysers, are not available [14]."
Hanke et al. 2013 [26][c]	4 (3%)	"In cases where the identification of plastic by visual inspection is ambiguous, i.e. for smaller items, confirmation might be sought by spectroscopy, e.g. FT-IR or Raman, or the "hot needle" technique may be employed." [in context of monitoring litter ingestion by fish, > 1 mm size]
Norén, 2007 [56]	4 (3%)	"In the laboratory, the particles showed the following properties: They were not dissolved in ethanol, acetone or xylene. The particles melted after having been heated gently on a microscope slide with a spirit burner. They emitted a distinct smell of plastics, as they were melting (smell of melted plastic rope end). Furthermore the plastic resolidified after having cooled and could easily be scratched with a sharpened glass needle." [in context of particles > 80 micron from water]

[a] Review paper

[b] Also found cited with the author as the Marine & Environmental Research Institute (MERI; Blue Hill, ME USA)

[c] Also found cited with the author as "Directive"

[d] Bellas et al. 2016 states "In case of doubt, a hot metal tip was applied on the object." [70] cites [18] and [26]

42] or a common sewing needle [29] heated with a "flame until red hot" have been described. A few studies specify using a candle or flame from a spirit lamp or (presumed butane) lighter [29, 32, 37]. Material and temperature are relevant parameters since synthetic polymers have a range of melting points. Time elapsed between heating and contacting the suspected microplastic under a microscope will impact the felt temperature as influenced by thermal conductivity of the needle. Only a couple of papers reviewed noted that the hot needle test may produce false negatives since thermoset plastics may not deform or melt at the temperatures applied [52, 57].

Across research studies, the hot needle test has been used as the sole verification of visual criteria or in combination with advanced polymer identification analytical tools. While some studies may only report microplastic counts that passed the hot needle test (e.g. [31], some others use it only on a subset of suspected microplastics as a confirmation step, or only when visual identification is tricky [1, 44, 72]. The hot needle test has been shown to be a helpful tool to distinguish some natural or anthropogenic particles (especially fibers) from microplastics. For instance, Welsh et al. 2022 [71] report that only 12% of 541 particles that they selected as visually appearing to be microplastics in atmospheric deposition samples actually melted, and they corrected their count data for this percent pass rate. When used in combination with other verification tools, the hot needle test is applied while counting under the microscope and a subset of suspected microplastics which pass are taken for advanced analysis, i.e. FTIR or Raman. However, the hot needle test has also been described as being used after spectroscopic analysis when results were uncertain (i.e. peak matching had a low hit quality index) [24, 41].

Confirmation rates of suspected microplastics that pass the hot needle test as synthetic polymers using spectroscopy vary widely in the literature. Korez et al. 2019 [40] analyzed beach sand for microplastics > 1 mm in size and counted them "if their shape changed upon contact with a hot needle", but only 11% were confirmed as

synthetic polymers by FTIR-ATR. The authors note that poor spectral matches were likely due to weathering, but that some were identified as dog fur, cashmere or wool. Among other studies of larger microplastics, Gholizadeh et al. 2022 [25] report a 76% confirmation rate by FTIR-ATR (> 200 micron, 30% subset isolated from sediment) with 15% being identified as non-plastic, and 9% unknown, while Lozano-Hernández et al. 2021 [45] report a 70% confirmation rate by micro-FTIR-ATR in their study (> 150 micron, 60% subset isolated from oysters) and Kapp et al. 2018 [34] report 67% confirmation (> 100 micron, from water samples) by Raman spectroscopy. Esiukova et al. [20, 21] in their recent studies report high confirmation (88 and 95%) of HNT-passed suspected microplastics (> 100 micron) by micro-Raman, but only 40% or 72% were actually confirmed against spectral matches, with the remaining being presumed microplastics due to the presence of strong fluorescence or dyes. However, it is important to note that other anthropogenic dyed materials, such as dyed cotton or semi-synthetic textiles (rayon, viscose) may also fluoresce and be difficult to identify by spectroscopy [2]. Klein et al. 2022 [38] analyzed by micro-FTIR 8% of suspected microplastics (> 50 micron) which passed the HNT and confirmed 32% as synthetic polymers, while 43% were dyed cellulose, and 19% were dyed unknowns. Jaafar et al. 2021 [33] analyzed a small subset of suspected microplastics ($N = 20$, 1.7% of total) by either FTIR-ATR or micro-Raman, depending on size, and report a 100% confirmation rate. High confirmation rate was also reported by Hurley et al. 2017 [31] using micro-FTIR-ATR ($N = 60$, particles > 50 micron), where all were confirmed except "a small number of fibers that could not be characterized using FT-IR as they were too small or transparent" but did "respond unambiguously to the hot needle test". Fibers are notoriously difficult to identify; Lutz et al. 2021 [48] analyzed a subset of suspected microplastics (> 63 micron) from sediment by micro-FTIR-ATR that passed the hot needle test and report a false positive rate of 51%, where the accuracy of identification was higher for synthetic polymer fragments (93% correct) than for fibers (22% correct).

Known fiber response to heat (melting point analyzer and soldering iron)

The synthetic fibers we tested, all semi-crystalline polymers, showed a softening or melt behavior in the melting point analyzer matching polymer reference data [12, 53] and showed responses to the hot point of a soldering iron that were described as melting, curling or shrinking (Table 3; see also videos linked in HNT 2 instruction sheet in Supplemental information). Acrylic softened but did not melt prior to decomposition in the melting point analyzer. Melting point (T_m) is the phase transition that occurs when the structure of the crystalline regions becomes disordered and starts to flow. Synthetic polymers can soften or become sticky at temperatures below their melting points. Amorphous polymers and the amorphous regions of semi-crystalline polymers exhibit a glass transition at a given temperature (T_g) or temperature range at which chain segments become more flexible and transition from a glass-like solid to a flexible state. T_g is below T_m, so it is possible that in some cases a softening of a semi-crystalline polymer will be observed due to a lower temperature or brief contact that brings a polymer to its glass transition but not its melting point.

The natural and semi-synthetic fibers showed only wavering (moving without phase change/alteration) or burning behaviors, with the exception of cellulose acetate from cigarette butt filters. While we observed several cigarette butt filter fibers to shrink or contract at ~ 220–240 °C in the melting point analyzer, at higher temperatures they burned but they could also melt. Cellulose acetate from cigarette butts has been reported to have a melting point of 240–243 °C (Benavente et al. 2018 [8]), similar to the shrink point described here. Cellulose diacetate has a glass transition temperature of 190–200 °C, but the form used in cigarette filters includes plasticizers as necessary for material processing and this can lower T_g considerably [5, 59]. "The burn test", used by sewers or garment workers to distinguish between different fabric's fiber content, does specify that acetate, when approaching a flame, will "blaze and burn quickly, then sputter, melt or drip like burning tar" [19]. Notably, the burn test cites some cellulosic fibers, like silk and wool, as "curling away from the flame", while synthetics tend to "shrink away from the flame". All cigarette butt filter fibers were observed to either move towards the soldering iron tip and stick, or to exhibit behavior that can be described as shrinking, softening or melting when contacted. When we brought the tip of the soldering iron near the cotton fiber, it showed a slight wavering behavior at every temperature in the ramp between 150 ℃ and 350 ℃. Responses by Spartina marsh grass were variable, and although not dramatic enough to likely be classified by an analyst as a positive test, we did occasionally observe a slight shrinking or curling of the ends of grass fibers. Cellulosic fibers can lose mass at lower temperatures (e.g. < 250 °C) due to loss of moisture and volatile chemicals (Cabrales & Abidi, 2010 [13]), which may explain wavering or slight curling behavior. Presence of volatile chemicals and thermal stability of natural and synthetic cellulose fibers can also change after digestion treatment, such as with alkali [65, 69].

Table 3 Fibers used in the present study with their melting (T_m) or burning points and observed behavior towards the hot point of a soldering iron set at 350 °C. Glass transition temperature (T_g) of polymers is provided from the literature

Synthetic textile fibers	T_g / T_m	Observed Response to Hot Point	Pass(+) or Fail(-) HNT
Polypropylene	-13°C[c] / 165–175 °C	Melting/curling	+
Acrylic	80–95°C[d]; 240–250°C[a]	Melting/curling	+
Polyester	70°C[a] / 240–260 °C	Melting/curling	+
Polyester-coated cotton	NA	Partial melting/shrinking	+
Nylon	50°C[c] / 270 °C	Melting/curling	+
Natural or Semi-synthetic	**Decomposition**[b]		
Cellulose acetate cigarette filter	190–200 °C (lit T_g) / 240–243 °C (lit T_m) 220–240 °C (shrinks); 250–300 °C (blackens); 270–280 °C (liquifies)	Melting/softening or possible wavering/shrinking/curling	+
Viscose	175–200 °C	No reaction	–
Silk	260–285 °C	No reaction	–
Cotton	210 °C	Wavering; movement away from heat; no curling nor melting	–
Spartina grass	NA		–
Digested, dead		Slight shrinking, burning	
Digested, live		No reaction	
Undigested, live		Slight curl or no reaction	

[a] A wide range of melting or softening points (~135–290 °C) for different types of acrylic fibers are found in reference guides [53]. We observed significant softening of the test material (sweater fiber) in the melting point analyzer at 240–250 °C prior to burning. Decomposition temperature of polyacrylonitrile is about 300°C[a]. Amorphous acrylics will not have a T_m

[b] Degradation of cellulose acetate from cigarette butt filters is observational data from a melting point analysis (ramp 10 °C/min) and a literature glass transition [5] for unplasticized cellulose diacetate) and melting point (Benavente et al. 2018) temperatures. Thermal decomposition temperature is reported in peer-reviewed and industry grey literature for viscose [51], silk [74] and cotton [49]

NA: Data not available

[c] Harper (2006) [27] for polypropylene, polyethylene terephthalate and Nylon (6,6)

[d] Richards [62] for polyacrylonitrile

Hendrickson et al. 2018 [29] also evaluated their method of bringing near suspected microplastics a glowing red-hot common sewing needle heated with a lighter. They report that their technique produced results on standards that were as expected (e.g. analytical plastic standards and polyester fibers melted, cotton and wool burned, and paint chips and Teflon did not respond).

Single-blind hot needle test trials

Based on the results of the melting point analysis and observed responses of our standards to a hot soldering iron, the correct responses to a hot needle test for our trials are shown in Table 3. Notably, we have classified cellulose acetate fibers from cigarette filters along with synthetic polymers as being able to pass the hot needle test, but another cellulosic semi-synthetic polymer, viscose, as not passing the hot needle test along with natural fibers.

For each material type, the percent of correct tests across 8 participants in HNT 1 are shown in Fig. 1 according to the hot point tool used (soldering iron at high heat 350 °C or low heat 150 °C, and a flame-heated needle, in this case either a hypodermic needle or metal dissecting probe heated to red hot with a butane lighter). The combined fraction of correctly identified fibers ($f_{correct}$) for each material type with statistics on whether results are significantly better than random guessing are reported in Table 4. Although we cannot rule-out an influence of the subjectivity of responses in HNT 1 since only 2–3 participants conducted the test by each of the separate methods, based on the results we observe that the soldering iron at the higher temperature setting performed best to identify synthetic polymers and cotton. All synthetic polymers were correctly identified by 4 of 8 participants (participant scores ranged 0.3–1, mean 0.8) and the aggregated results indicated that the HNT aided identification (nylon $\chi^2=12.25$, $p=0.0005$; polyester $\chi^2=5.76$, $p=0.02$; acrylic $\chi^2=8.05$, $p=0.005$). Cigarette filter fibers were also more likely to be identified as passing using the high temperature soldering iron in comparison to the other techniques, with the undigested fibers being identified as passing at a higher rate than digested fibers ($f_{correct}$ 0.64 versus 0.22, respectively). The flame-heated needle also performed well for several fiber types (nylon,

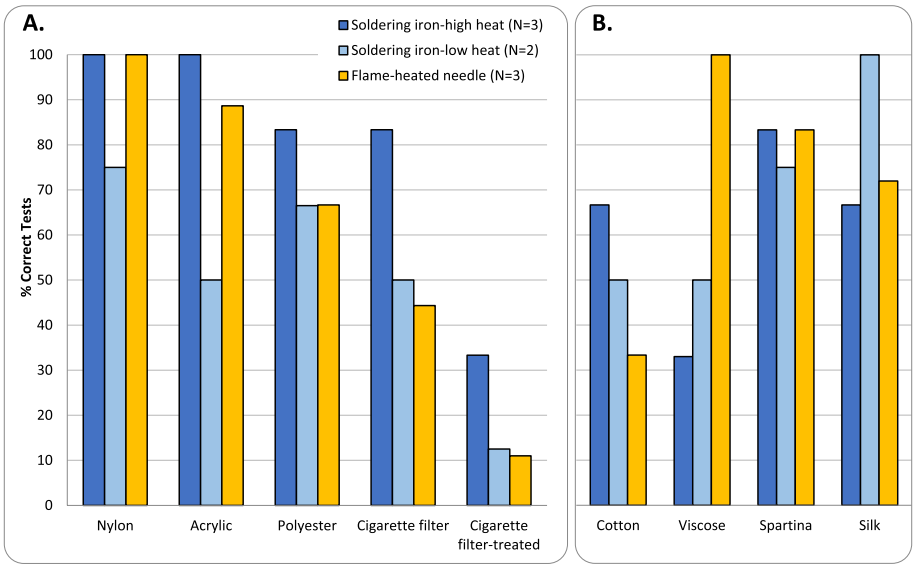

Fig. 1 Percent correctly identified by each probe type (soldering iron at high heat 350 °C, soldering iron at low heat 150 °C or flame-heated needle) for each fiber type according to expected response, with materials expecting to pass the hot needle test in A) synthetics and cellulose acetate in cigarette filters and fail in B) semi-synthetic and natural fibers. The "treated" cigarette filter fibers were digested in 1 M KOH for 72 h. Total % correct equally weights participant scores (i.e. does not account for differences in numbers of fibers tested across individuals)

Table 4 Chi-square goodness of fit test whether the proportion of correct or incorrect fiber tests is significantly different than 0.5 (alpha = 0.05, DF = 1, critical value = 3.84, *N* = number of fibers tested across all individuals performing the test). Fraction of correct fibers tested ($f_{correct}$) is also tabulated

Material	HNT 1				HNT 2			
	$f_{correct}$	χ^2	*p*	N	$f_{correct}$	χ^2	*p*	N
Nylon	0.94	12.25	**0.0005**	16	1.00	11.00	**0.0009**	11
Polyester	0.76	5.76	**0.02**	21	0.79	4.57	**0.03**	14
Acrylic	0.81	8.05	**0.005**	21	0.42	0.33	0.56	12
Cigarette filter	0.64	1.64	0.20	22	0.57	0.29	0.59	14
Cigarette filter-digested	0.22	7.35	**0.007**	23	0.55	0.09	0.76	11
Polypropylene	NA				1.00	15.00	**0.0001**	15
Spartina grass (dead)	0.81	6.25	**0.01**	16	NA			
Cotton	0.50	0.00	1.00	18	0.82	7.12	**0.008**	17
Viscose	0.65	2.13	0.14	23	1.00	17.00	**0.0001**	17
Silk	0.76	4.76	**0.03**	17	1.00	12.00	**0.0005**	12

HNT 1: participants use their preferred method

HNT 2: all participants use a soldering iron at 275 °C with test criteria guidance

NA: Not applicable; material wasn't used in the test

acrylic, viscose). The low percent correct identifications of cotton were of concern (i.e. 1/3, 1/5 and 2/3 of participants incorrectly recorded that cotton fibers passed for the soldering iron at high and low temperature and the flame-heated needle, respectively, $f_{correct} = 0.50$), considering it is a natural fiber that should have no melt behavior. We did observe a wavering movement of cotton fiber during the temperature ramp testing (150 ℃-350 ℃), which could possibly be attributed to a loss of moisture and volatile chemicals from the fiber. However, we emphasize that the waver reaction does not fit into the required reaction criteria to be considered a microplastic. Participant scores for fibers not expected to pass the hot needle test (natural fibers and viscose, listed in Table 3) were lower on average than for synthetics (ranged 0.38–1, mean 0.68). The hot needle test

only aided identification of spartina grass and silk significantly (spartina χ^2=6.25, p=0.01; silk χ^2=4.76, p=0.03). Higher rate of misidentification is likely due to the lack of standardization in the hot needle test training in the first trial.

The second hot needle test trial aimed to assess the method when participants use the same tool at the same temperature and are given specific guidance regarding what constitutes a positive pass response. The criteria provided is shown in Table 1 (see also Supplemental Information).

Comparison of the percent correct tests across individuals for each material type in HNT 1 and 2 is shown in Fig. 2 (individual-level results are provided in Supplemental information Table S2; $f_{correct}$ and Chi-square goodness of fit test results in Table 4). Performance was similar for synthetic fibers except acrylic. For HNT 2, the soldering iron was set to 275 °C which is above the softening point observed for our acrylic material (polyacrylonitrile) and below its decomposition temperature (Table 3). With brief contact, the acrylic fiber became sticky and discolored/browned. Softening of acrylic was much more evident with longer contact of the probe (a few seconds) or at higher temperature (350 °C, as seen in Table 3). We therefore expect that the variable result for acrylic in HNT 2 was due to differences in contact time and interpretation among participants. These results are also supported by HNT 1; the 350 °C setting was 100% accurate to identify acrylic, whereas the 150 °C soldering iron was only 50% accurate. Correct identification of cotton (83%), viscose (100%), and silk (100%) improved in HNT 2 (Fig. 2, Table 4). Four individuals participated in both HNT 1 and HNT 2; in HNT 1, one used a flame-heated needle, one used the high temperature setting and two used the low temperature setting on the soldering iron. One participant using the low temperature setting in HNT 1 correctly identified every natural fiber in both trials. Since marked improvements were seen in correct identification of natural fibers for each of the other three participants in HNT 2 compared to HNT 1 (Fig. 2, Table 4), we conclude that it was training and guidance on what constitutes a "pass versus fail" reaction, and not the selection of tool that was most important. The percent pass rate of digested cigarette filter fibers was also higher in HNT 2 than HNT 1, although the rate was still relatively low (~50% identified as passing). When testing these fibers with the soldering iron at 275 °C, we did observe that the smaller the fiber, the more difficult it was to assess the response. The hot needle test, as performed in this study, is therefore unreliable for identifying cellulose acetate from cigarette filters due to the subjectivity of analysts considering the variability in material response to heat (Table 3).

Raman spectroscopy analysis of hot point-tested environmental samples

We analyzed two different subsets of suspected microplastics (>63 micron) by micro-Raman spectroscopy to evaluate performance of the hot point test that was conducted with a soldering iron using specific response

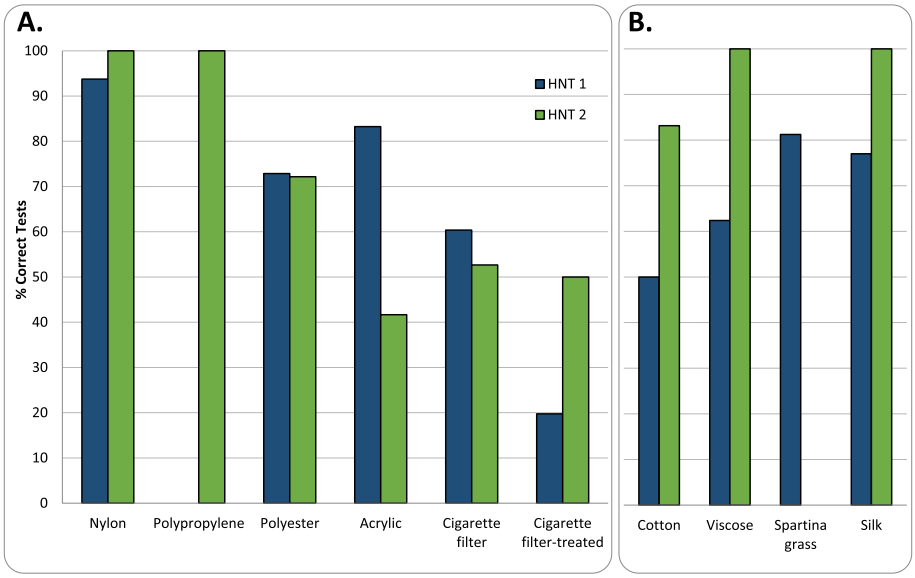

Fig. 2 Percent of correctly identified fibers in HNT 1 (N=8 participants, different techniques combined) and HNT 2 (N=6 participants, all use the same soldering iron technique) by material type according to expected response, with materials expecting to pass the hot needle test in A) synthetics and cellulose acetate in cigarette filters and fail in B) semi-synthetic and natural fibers

criteria (see Table 1). In the first set from pond water, there was a 100% confirmation rate of all suspected microplastic fibers as synthetic polymers as well as those expected not to be synthetic. Microplastic fibers ($N=8$) were polyester (50%), nylon, polyurethane, polyethylene and acrylic. Microfibers that did not pass the hot needle test ($N=7$) were correctly identified as organic materials in the spectral library and included waxed cotton, dyed cotton, cellulose, or were burned at low laser intensity in which case the material was presumed to be organic matter. In set 2 ($N=63$), there was a 90.5% confirmation of synthetic polymers. Several spectra were unresolved due to fluorescence (4.8%) and one was tentatively identified as cellulose acetate (1.6%) which as mentioned above is a semi-synthetic polymer that is common in cigarette butts, an abundantly littered item in urban environments and can respond positively to the hot needle test. In these urban sediment samples, microplastics were predominately polypropylene (38%) and polyethylene (35%), with additional detections of polyurethane, polyamide, polystyrene and polyvinyl chloride.

Conclusions

The hot needle test, when implemented carefully, is an affordable and complementary tool for microplastic identification. For the HNT to work well and result in few misidentifications, clear guidelines with descriptions of the response to a heated probe are needed for common microplastics as well as natural, anthropogenic and semi-synthetic fibers that are present in samples. The following reaction criteria are recommended (Table 1):

1) *A positive result* shows a melt, softening or significant bend/curl behavior; and
2) No response or a movement/waver behavior with no shape change is *a negative result*.

Different heated metal implements can be used, but we have found a soldering iron to be practical. It is important to test the function of the hot point selected at the beginning of the project against standards with known melting points, and over time to check for consistency in the heat source; for instance, a sand bath or soldering iron may need thermostats replaced, and metal tips may oxidize over time resulting in loss of heat transfer capability, requiring them to be cleaned or replaced periodically. If strong digestion reagents are used in sample processing steps that degrade a polymer significantly [66] and alter its thermodynamic properties (e.g. T_m, T_g), then an altered response to the hot needle test would be expected, but this is an area for future study. The hot needle/hot point test is only one verification step that is useful for confirming identity of a subset of microplastic polymer types. It is advisable that some additional confirmation of the HNT results be performed, such as by spectroscopy, to provide higher confidence in correct classification of suspected microplastics with the added benefit of providing polymer characterization information. For instance, synthetic coatings on natural fibers can influence HNT results and, while arguably not microplastics, it is interesting to be able to report such anthropogenic materials since the synthetic coatings may have implications for material fate and toxicity. Additional method development to identify cellulose acetate fibers from cigarette butts, which are one of the most found litter items globally, is warranted given their potential abundance and low degradability as well as their ecotoxicological concern [7]. Impact of variable detection of cellulose acetate fibers depends on the intention of a study to count strictly synthetic microplastic fibers or anthropogenic fibers including semi-synthetic bioplastics. As a part of quality assurance/control procedures, researchers should test their ability to differentiate between synthetic and natural polymer materials common to their matrices and within their size class of interest. We offer an example of a single-blind trial to evaluate the HNT that may be adapted by other laboratories.

Abbreviations

HNT	Hot needle test
FTIR-ATR	Fourier transform infrared spectroscopy-attenuated total reflectance

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s43591-023-00056-4.

Additional file 1:Table S1. Top 20 Journals in Web of Science "microplastic*" by hit number results (June 2022): top 9 are included in the present literature review. **Table S2.** Individual-level summary results of single-blind microplastic fiber identification trials using the hot needle test (trials #1 and #2). Expected reactions by fiber type are provided in main text. Values are fraction of fibers that were correctly identified. Total % correct equally weights participant scores (i.e. does not account for differences in numbers of fibers tested across individuals). Additional file also includes survey instructions and data sheets for single blind trials of hot needle test, and complete systematic literature review reference list.

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Authors' contributions

CRediT statement: BB: Conceptualization, Methodology, Investigation, Resources, Data Curation, Writing – Original Draft, Supervision, Funding acquisition. AA: Methodology, Investigation, Formal analysis, Data Curation, Writing – Original Draft, Funding acquisition. CM: Formal analysis, Writing

– Review & Editing. JB: Resources, Writing – Review & Editing. All authors read and approved the final version of the manuscript.

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Availability of data and materials
All data generated or analyzed during this study are included in this published article and its supplementary information file.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare that they have no competing interests.

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