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Self-identification of nonpharmaceutical fentanyl exposure following heroin overdose

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Abstract

Objective—To compare user self-identification of nonpharmaceutical fentanyl exposure with confirmatory urine drug testing in emergency department (ED) patients presenting after heroin overdose.

Methods—This was a cross-sectional study of adult ED patients who presented after a heroin overdose requiring naloxone administration. Participants provided verbal consent after which they were asked a series of questions regarding their knowledge, attitudes and beliefs toward heroin and non-pharmaceutical fentanyl. Participants also provided urine samples, which were analyzed using liquid chromatography coupled to quadrupole time-of-flight mass spectrometry to identify the presence of fentanyl, heroin metabolites, other clandestine opioids, common pharmaceuticals and drugs of abuse.

Results—Thirty participants were enrolled in the study period. Ten participants (33%) had never required naloxone for an overdose in the past, 20 participants (67%) reported recent abstinence, and 12 participants (40%) reported concomitant cocaine use. Naloxone was detected in all urine drug screens. Heroin or its metabolites were detected in almost all samples (93.3%), as were fentanyl (96.7%) and its metabolite, norfentanyl (93.3%). Acetylfentanyl was identified in nine samples (30%) while U-47700 was present in two samples (6.7%). Sixteen participants self-identified fentanyl in their heroin (sensitivity 55%); participants were inconsistent in their qualitative ability to identify fentanyl in heroin.

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Disclosure statement

No potential conflict of interest was reported by the authors.

Conclusions—Heroin users presenting to the ED after heroin overdose requiring naloxone are unable to accurately identify the presence of nonpharmaceutical fentanyl in heroin. Additionally, cutting edge drug testing methodologies identified fentanyl exposures in 96.7% of our patients, as well as unexpected clandestine opioids (like acetylfentanyl and U-47700).

Keywords

Heroin; fentanyl; overdose; acetylfentanyl; U-47700; opioid epidemic; drug testing; time-of-flight

Introduction

The amount of fentanyl required to kill a 70 kg adult is equivalent to a few grains of salt; even less is needed to produce lethal overdose once mixed with illicit opioids, such as heroin. The adulteration of heroin with nonpharmaceutical fentanyl is not new [1–3]. What is new, however, is the broad geographical scale of heroin adulteration with nonpharmaceutical fentanyl, and the number of other distinct, high-potency, clandestine opioids being added to heroin [4–6]. Little is known about the incidence, identity and clinical impact of these other clandestine opioids. In this analysis, we conducted a cross-sectional study comparing user self-identification of nonpharmaceutical fentanyl exposure with rigorous drug testing designed to identify both fentanyl and other clandestine opioids in a cohort of patients who presented after heroin overdose.

Nonpharmaceutical fentanyl is hypothesized to fuel the striking increase in heroin overdose deaths [7,8]. Indistinguishable from pharmaceutical material, nonpharmaceutical fentanyl produces nearly immediate onset of opioid effects, including cessation of respiratory effort [1]. The widespread adulteration of heroin in the United States and Canada with nonpharmaceutical fentanyl is accompanied by increasing reports of other clandestine opioids identified in postmortem casework and product seizures [9–11]. Some of these (denoted fentanyls in Table 1) are congeners of fentanyl; others (like U-47700) are structurally distinct. Both fentanyl and nonfentanyl clandestine opioids are mu-opioid receptor agonists; several are more potent than heroin, resulting in an increased risk of respiratory depression after use [9–11]. Experts postulate that the addition of nonpharmaceutical fentanyl and other clandestine opioids to heroin increases overall potency, making the product more desirable and marketable. Within the last two years, there has been a sharp increase in the number of new clandestine opioids that have been identified [12].

Self-report, normally an efficient method for assessing dimensions of drug use, may fail in these cases as drug users are unaware of heroin adulteration. Moreover, the tiny doses of nonpharmaceutical fentanyl or clandestine opioids needed to produce poisoning are frequently insufficient to alter the color or consistency of heroin and make them indistinguishable to users [13]. Despite their pharmacologic similarity to other opioids, the clandestine opioids have different chemical structures that may not produce positive results on traditional drug screens for opioids [14]. In several cases, negative opioid testing in patients with signs of injection drug use ultimately led to the diagnosis of clandestine opioid toxicity [15–17].

Rigorous testing for fentanyl and other clandestine opioids is not routinely available for clinical use, and the methods described represent an innovation from traditional urine drug testing for opioids that can be used to guide surveillance and public health interventions.

Our objectives in this pilot study were to: (1) compare heroin users' self-identification of nonpharmaceutical fentanyl exposure with urine drug testing results; (2) describe user beliefs regarding differences in heroin appearance, preparation, or effect that distinguished the presence of non-pharmaceutical fentanyl; and, (3) identify the presence of other clandestine opioid exposures in our population.

Methods

Study design

We performed a cross-sectional study of adult patients who presented after a heroin overdose requiring naloxone for the reversal of respiratory depression. Participants completed a semistructured interview concerning knowledge, attitudes and beliefs regarding heroin and nonpharmaceutical fentanyl; participants also provided a urine specimen for drug testing. Verbal consent was obtained from participants; survey answers and drug testing results were collected in an anonymized fashion. The University of Massachusetts Institutional Review Board hospital approved the study protocol.

Study setting and participants

We conducted this study at an urban, medical school-affiliated adult ED in New England. Both the adult and pediatric EDs are level one trauma centers, providing medical care to greater than 80,000 patients per year.

Throughout the study period, three physicians and a research assistant (RA) enrolled patients during preselected dates/times of enrollment. During enrollment periods, the charts of all patients presenting to the ED after naloxone administration for suspected heroin overdose were screened for eligibility. Eligible patients, as determined by chart review, were then approached in person for further screening. Eligible patients were >18 years of age, English speaking and able to provide informed consent. Patients were excluded if they were prison inmates, critically ill, or unable to provide consent. No incentives were offered to participants.

Interviews

Study-eligible ED patients were asked to participate in a 15-min semistructured interview, consisting of brief demographic information (age, sex, ethnicity, race) and open-ended questions regarding their drug use. The interview prompts were as follows: (1) medications used in the 72 h prior to overdose (with a focus on fentanyl by prescription); (2) illicit drugs used in the 72 h prior to drug use; (3) routes of administration for illicit drugs; (4) history of overdose; (5) intent to purchase/use heroin; (6) intent to purchase/use fentanyl; (7) perception that clandestine fentanyl was present in the drugs used prior to overdose; and, (8) last period of abstinence. We used an interview guide for this portion of the survey to ensure that all study investigators asked the same questions in the same format. Responses were

recorded in real time and transferred to a spreadsheet. The responses from the semistructured survey were transcribed to create a dataset for thematic analysis. This dataset was intended to obtain formative information related to heroin use. We used an abstraction form to collect information. Survey responses were reviewed by two independent investigators until thematic saturation was achieved; conflicts were resolved by a third reviewer to produce a summary of identified themes.

Specimen handling

Urine collection occurred at the time of enrollment. After survey completion, subjects were asked to provide an unsupervised urine specimen. A minimum of ten milliliters of urine was required for analysis. All specimen cups were labeled with a bar code/number that linked the urine specimen to the survey. The specimens were placed in a second impermeable bag and stored at 25 °C until shipping. Samples were shipped via overnight mail to the Center for Forensic Science Research and Education once weekly, where they were stored at 4 °C and analyzed within one week of collection.

Urine drug testing

All testing was performed at the Center for Forensic Science Research and Education (Willow Grove, PA). In addition to the clandestine opioids listed in Table 1, the urine specimens were tested for the presence of other opioids (e.g., hydrocodone, oxycodone, methadone, oxymorphone), heroin metabolites (e.g., 6-MAM, morphine, codeine), common pharmaceuticals and drugs of abuse, totaling more than 400 compounds. Specimens were prepared via a single-step basic (pH 10.4) liquid–liquid extraction, a protocol previously validated for clinical and forensic work [18]. Verification was performed using clandestine opioid standards (including fentanyl, norfentanyl, acetylfentanyl, carfentanil and U-47700) in extracted and unextracted forms, from applicable biological specimens. Sensitivity studies were conducted for the determination of threshold detection limits: fentanyl 1 ng/mL, norfentanyl 2 ng/mL, acetylfentanyl 1 ng/mL, carfentanil 1 ng/mL and U-47,700 1 ng/mL.

Analysis was performed by liquid chromatography quadrupole time-of-flight mass spectrometry (LC-QTOF-MS) using a SCIEX TripleTOF® 5600 + QTOF and a Waters Xevo® G2-S QTOF. Data processing was performed against extensive in-house databases that include exact mass, retention time, exact fragment masses and library spectra. Positive drug identifications were made based on pre-established criteria, consistent with industry standards. Comparison of results by platform was conducted for confirmation. During this analytical process, we were able to identify the presence of fentanyl and clandestine opioids with a high degree of specificity.

Data analysis

Two of the study coinvestigators independently reviewed the transcribed comments of participants. Before conducting their review, the investigators underwent a training session with senior investigator to standardize the review protocol. Each participant’s semistructured interview responses were categorized according to the following hierarchy: (1) did the participant respond “yes,” “no,” or “I don’t know” to the question regarding self-identification of fentanyl exposure; (2) if the participant responded “yes” to suspected

fentanyl exposure, did s/he describe distinguishing features in the appearance, preparation, or effect of the drug (these were not mutually exclusive). The investigators agreed a priori that a third arbitrator would mediate if consensus could not be achieved, but there were no cases for which this was required.

The presence of various drugs is reported as raw number and percentage throughout. Agreement between self-identification and presence of fentanyl in urine is reported as a sensitivity and Cohen's kappa index value. Agreement between coders for qualitative themes is also reported as a kappa statistic.

Results

We enrolled the study participants between 24 August 2016 and 11 December 2016. Over this time, 69,350 patients were treated in the ED; during our preselected enrollment periods, 55 received a diagnosis of heroin overdose and were considered eligible. During the study period, 32 eligible individuals were approached for consent; during this pilot study, we did not have overnight coverage for enrollment. Two individuals declined participation. Thirty individuals consented to study participation (93.8%). The demographic characteristics of participants can be found in Table 2.

Drug use history

All of the enrolled participants reported heroin use, with injection use by 22 (73.3%), intranasal use by seven (23.3%) and one individual reporting use by both routes (3.3%). Ten (33%) denied any prior overdoses requiring naloxone administration. All of our participants reported an intent to purchase/use heroin; all denied seeking fentanyl. Twenty participants (67%) self-reported recent abstinence, a known risk factor for overdose.

Study participants reported high rates of concomitant medication and drug use. Participants reported use of antidepressants ($N = 9$, 30%), anticonvulsants ($N = 6$, 20%) and benzodiazepines ($N = 8$, 26.6%) in the 72 h prior to their ED presentation. Two patients reported buprenorphine (Suboxone) use, while one endorsed methadone use. Additionally, 12 (40%) participants admitted to concomitant cocaine use; only one reported methamphetamine use. No patients were prescribed fentanyl, or reported using fentanyl prior to ED presentation.

Urine drug testing results

Naloxone was detected in all of the urine specimens. However, heroin and heroin metabolites (6-monoacetylmorphine, morphine and codeine) were found in three (10%), 20 (67.7%), 27 (90%) and 24 (80%) of the urine specimens, respectively. Two participants in this study tested negative for heroin and metabolites, despite reporting its use. Fentanyl and its metabolite, norfentanyl, were found in 29 (96.7%) and 28 (93.3%) of our participants' urine specimens. Nine of the participants in this study used acetylfentanyl, while two patients (who were enrolled several days apart) were exposed to U-47700. In addition, we identified prescription opioids and benzodiazepines (Table 3). Five participants tested positive for methadone, while four were positive for buprenorphine. Detailed urine testing results can be found in Supplementary Table 1.

Overall, we found more drug use by urine drug testing than participants self-report. Regarding other classes of drug exposure, 20 participants had cocaine identified in urine, with 17 positives for benzoylecgonine (a cocaine metabolite); 21 patients were positive for levamisole (a common cocaine adulterant). Five participants were methamphetamine positive. A comparison of self-report of drug use and urine testing can be found in Table 3.

Self-identification of nonpharmaceutical fentanyl exposure

There was 100% agreement between two investigators in coding the responses to the hierarchy described earlier. Of the 29 participants who had fentanyl identified by urine testing, 16 positively self-identified their fentanyl exposure; nine answered “I don’t know”, while four denied fentanyl exposure. The one participant who tested negative for fentanyl exposure correctly self-identified exposure to heroin only (Table 4).

The sensitivity of self-identification of fentanyl exposure in participants when compared with a gold standard of urine drug testing was 55%, with a calculated Cohen’s kappa index value of .076. Qualitative comments about the perceived differences between heroin and fentanyl are reported in Table 5. Of the participants who correctly identified their non-pharmaceutical fentanyl exposure, the realms of the distinguishing characteristics are listed in Table 6.

Discussion

We detected the presence of nonpharmaceutical fentanyl in nearly all of the tested urine specimens (96.7%). The sensitivity of self-report of suspected nonpharmaceutical fentanyl exposure, however, was only 55%. In two participants who presented after self-reported heroin overdose, no heroin metabolites were detected in their urine. Anecdotally, many heroin users have reported that fentanyl can be identified by its lack of color, or white appearance. Of the fentanyl-exposed participants, three characterized their drug as white or colorless, while two others described a yellowish/brown color; others noted no difference in the appearance of the drug at all. Six heroin users in this study identified a distinct effect on using a fentanyl-adulterated drug; this finding is particularly frightening since the point of user recognition did not allow time to get help or treatment prior to the impending overdose.

In this study, we also identified the presence of unexpected clandestine opioids in a cohort of patients presenting to an emergency department after heroin overdose, including acetylfentanyl (30%) and U-47700 (6.7%); both participants who tested positive for U-47700 also tested positive for acetylfentanyl exposure. We did not identify the presence of carfentanil our study cohort. Explanations for this finding include: (1) carfentanil produces clinical effects at ultra-low (pg/mL) concentrations that may fall below the threshold of detection and more likely; (2) the absence of carfentanil in our catchment area, as carfentanil has not yet been documented in Massachusetts. Our results support existing literature describing the emergence of new clandestine opioids as a significant public health problem, and indicate the need for systematic drug testing surveillance strategies to improve upon unreliable user self-report and to inform public health strategies for mitigating opioid overdose [19–21].

Our data contrast with recent state level data from Massachusetts. Of the 693 individuals with opioid-related deaths in Massachusetts in 2016 (and concomitant toxicology screening), 510 (74%) were positive for fentanyl. During the second quarter of 2016, heroin, or likely heroin, was identified in only 53% of opioid-related deaths where toxicology results were available [22]. Additionally, although the increased presence of heroin and fentanyl in our population may be due to our improved detection methods, the difference suggests that post-mortem data alone may be insufficient to guide patient care.

Overall, the heroin users in this study underreported concomitant drug use when self-identification was compared to urine drug testing. It is interesting to note that five of the participants in this study had evidence of buprenorphine use. Although the timing of buprenorphine use remains unclear, the presence of high-potency opioids raises the possibility that clandestine fentanyl use may be sufficient to overcome the partial agonist effect of buprenorphine. Interestingly, none of the participants in this study reported a tramadol exposure, yet urine drug testing identified seven (23.3%) participants who had used tramadol.

One striking feature of this study was the willingness of a high proportion of patients to participate in this uncompensated effort so soon after reversal of their overdose. An overarching theme of participation was the desire “to do something” to help prevent overdose deaths. One participant reported that she was participating because she had lost three friends to overdose in the prior month. Another dramatic finding was that nearly 50% of study participants were women, in contrast to national data that demonstrates a male predominance among heroin users [23].

Limitations of this study include recall bias when asking heroin users about prior 72-h drug and medication use. Urine drug testing identifies nonpharmaceutical fentanyl and clandestine opioid use during approximately the prior 72 h [24]. In one study of seven surgical patients who received fentanyl intraoperatively, 100% had detectable fentanyl in urine postoperatively using GC/MS with a detection threshold of 0.1 ng/mL. That proportion declined to 42.3% at 12 and 24 h, 14.2% by 48 h and 0% by 72 h [24]. As a result, we cannot specifically point to nonpharmaceutical fentanyl or a clandestine opioid as the culprit agent contributing to acute overdose; the presence of these drugs in urine may be the residua of prior use within the detection window that did not result in overdose. Additionally, we did not collect data on naloxone dosing required for reversal in this work, which allowed us to obtain verbal consent only in a truly anonymous fashion.

Moreover, our results are unlikely to reflect other populations and settings outside of this region, and cannot be easily generalized to other EDs with patients of different demographic distributions. However, our methodology can be employed by other institutions to establish local patterns of nonpharmaceutical fentanyl and clandestine opioid use.

Further research is urgently needed to explore the prevalence of clandestine opioids in overdose, the pharmacokinetics of these novel agents and strategies to prevent overdose in patients who are unknowingly exposed to these high-potency opioids. One solution may be

"sentinels," or pre-designated emergency departments with protocols for standardized surveillance of clandestine opioids using this methodology.

In summary, over 96% of the participants in this study had a nonpharmaceutical fentanyl exposure; however, only 55% were able to accurately self-identify this exposure. These data are significant because a lack of ability to self-identify fentanyl limits the development of interventions targeting fentanyl recognition in heroin users who overdose and demonstrates the significant challenges posed in determining whether fentanyl is a major contributor in individuals who present after a suspected opioid overdose. Using this methodology, we also found two unexpected clandestine opioids (acetylfentanyl and U-47700) in the urine specimens provided by the heroin users in this cohort. Cutting edge drug-testing methodologies can assist clinicians in better characterizing the drug use habits of their population, as well as the emergence of novel clandestine opioids.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Table 1

Clandestine opioids.

Fentanyls
Acetylfentanyl
Alpha-Methylfentanyl
3-Methylfentanyl
Butyrylfentanyl
p-Fluorofentanyl
Beta-Hydroxythiofentanyl
Valerylfentanyl
Furanylfentanyl
Carfentanil
Norfentanyl (metabolite)
Nonfentanyls
U-47700
U-50488
AH-7921
MT-45

Table 2

Demographic characteristics of participants.

	***N* = 30**
Median age, years (IQR)	20 (17–23)
	%
Sex	
Male	45.4
Female	54.6
Ethnicity/Race	
White, non-Hispanic	86.7
Black, non-Hispanic	0
Hispanic	13.3
Other	0

Table 3

Comparison of self-report versus urine presence of specific drugs ($n = 30$).

Drug	Self-report	Parent compound or metabolites confirmed in urine
Heroin	30	28
Fentanyl	16	29
Norfentanyl	N/A	28
Acetylfentanyl	0	9
U-47700	0	2
Cocaine	12	20
Methamphetamine	1	5
Methadone	2	5
Buprenorphine	1	5
Tramadol	0	7
Oxycodone	0	6
Hydrocodone	0	5
Alprazolam	3	6
Clonazepam	6	11
Lorazepam	0	2

Table 4

Self-identification of nonpharmaceutical fentanyl exposure versus urine drug testing results.

	Urine drug testing for fentanyl	
	Positive	**Negative**
Self-Report of Fentanyl Exposure		
Yes	16	0
No	13	1

Sensitivity 55%, Cohen's kappa index value 0.76.

Table 5

Selected comments from semi-structured interviews from participants; all had fentanyl identified in urine.

Self-identification		Phase of use
Yes	“I know it was fentanyl, it was white, colorless-no odor to it. It felt different”	Appearance/Effect
	“Yes - I never had heroin like this. This heroin was light brown in color, I got it from a person I bumped into.”	Appearance
	“Yes - I could tell when I dumped it out. It was smooth up the nose. It didn’t burn. It felt like a ‘pill’ not a vinegar taste.”	Appearance/Effect
	“I had a feeling it had fentanyl in it, used it yesterday and knew it was too strong/good.”	Effect
	“I think this was stronger.”	Effect
	“The powder was light in the bag, but darkens when dissolved in water with residue left over in the cap.”	Appearance/Preparation
	“I’ve never gone out like that before. It looked funny - like sand - and it didn’t suck up right away - there was residue on the spoon, I had to cook it twice.”	Appearance/Preparation/Effect
	“Yes, without a doubt. It was white. As soon as I shot up, it was like boom, right up.”	Appearance/Effect
I don’t know	“I guess it was stronger. It all looks the same to me.”	
	“Maybe, I don’t really know though. I just remember injecting and nothing else.”	
No	“Nothing different about the color, texture, or taste.”	

Table 6

Distinguishing characteristics described by participants who correctly identified fentanyl exposure ($n = 16$).

Phase of use	
Appearance	1 (6.3%)
Effect	3 (18.8%)
Appearance + Preparation	2 (12.5%)
Appearance + Effect	6 (37.5%)
Appearance + Preparation + Effect	2 (12.5%)
None of these	2 (12.5%)
Total	16