

# Research Article

## Oil Composition of Pilis: An Indonesian Forehead Topical Herbal Medicine

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### ABSTRACT

**Context:** Pilis is a type of jamu (Indonesian herbal medicine) applied to the forehead to cure dizziness, hazy vision and eyestrain. At the moment, pilis can be obtained from large jamu factories, local jamu producers, and home-based producers. Information on the diversity of pilis quality from producers is needed to determine the most essential bioactive compounds in Pilis. **Aims:** This study was undertaken to compare the phytochemical compositions of several pilis oils produced by large jamu factories, local jamu producers, and home-based producers by GC-MS chemical analysis. **Material and Methods:** Pilis oils were sourced from multiple local manufacturers. All oils were extracted with hexane by Soxhlet extraction. The crude extracts were analyzed by GC-MS. **Results:** The oil composition of various pilis samples was complex and diversified. Many constituents were identified. The oil constituents of pilis could be classified into essential oils, fatty acids, and other nonpolar compounds. Pilis from the large factory had the lowest essential oil content, whilst the pilis from home-based producer was the highest essential oil content. Twenty three main essential oils were detected. The amount of fatty acids in the pilis sourced from large factories was the highest. The fresh made pilis from home-based producers had a smaller amount of fatty acids than others. The other nonpolar constituents detected in all pilis were dimethoxyphenyl butadiene (DMPBD), shogaol and amyirin. **Conclusion:** Fresh made pilis from home-based producers had a higher amount of essential oils and also had higher variation, but only a small amount of fatty acids compared to the pilis from large jamu factories as well as local jamu producers.

**Key words:** Essential oils, Jamu, Pilis, Topical Herbal Medicine, Indonesian traditional medicine, Ethnobotany.

### INTRODUCTION

Jamu is made from Ramuan, a conventional Indonesian herbal blend supported by centuries of use. Ramuan means synergetic blending. Pilis is a type of Jamu (herbal medicine) applied to the forehead to cure dizziness, hazy vision and eyestrain, and blurred eye visions. As a medicated talcum paste, pilis is applied onto the forehead. Pilis is also often used for children who have fever as an antipyretic medicine. It is believed this treatment also helps to prevent headache during old age. It is usually used after pregnancy which is a time of great physical and emo-

tional change for a woman. It is also believed to have anti-stress activity. Pilis originates and is well known in Java, Indonesia, but now not many women use it after-birth. Together with tapel (paultice), param (medicated talcum paste applied to the whole body) sembur (chewed herbal materials applied to forehead or scalp) and bobok (medicated talcum paste applied to certain part of the body), pilis belongs to the transdermal or topical herbal medicine. Tapel is used to help to tone and tighten up the stomach muscles and expel wind out from the tummy after delivery. Sembur is done by the Dukun, a traditional healer, who chews the plant/herb materials until becoming fine and then spittle or spray directly to the forehead, scalp or stomach. Bobok or jamu hangat (warm Jamu) is used for cure pegal (muscle strain). The objective of this study was to compare the oil composition of pilis from different producers: large producers, local pro-

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ducers, and home-made producers. The analysis was performed by GC-MS.

## MATERIALS AND METHODS

### Samples

Pilis were obtained from various producers either from large jamu industries, local jamu industries, or home-made jamu producers. The total number of samples analyzed was 16 samples. Large jamu factories are well-known (Air Mancur, Jago, Meneer), while local jamu producers can only be found in certain cities. Both types of pilis come in a package or small plastic/paper bag in dry condition with a trademark. The home-based industry pilis had no trademark, was covered with banana leave and sold in the local market in a wet/fresh condition. Several home-based products were sourced from different manufacturers using fresh plant materials. These materials were mixed, crushed and pounded until a paste was obtained.

### Oil Extraction with Hexane

Prior to the extraction, the samples of pilis were crushed and powdered or pounded till powders were obtained. A mass of 100 grams pilis powder was extracted with n-hexane using Soxhlet extraction exhaustively for 8 hours. The hexane extracts were concentrated under reduced pressure to provide a concentrated extract. The concentrated hexane extract obtained was submitted for GC-MS analysis.

### Chemical Analysis with GC-MS

GC-MS analysis was performed on an Agilent Gas Chromatography Model 6890 coupled to an Agilent 5973 Mass Selective Detector. Analytes were separated on an HP 5MS capillary column (60 meter x 0.25 mm x 0.25  $\mu$ m). The following temperature program was applied. Oven initial temperature was 100°C (On) and maximum temperature was 350°C. Initial time was 0.00 min equilibrium time was 0.50 min. The initial front inlet (split) was 290°C. Pressure was 20.90 psi, split ratio was 50:1, split flow was 49.8 ml/min, total flow was 53 ml/min, and gas type was Helium. Compound identification was done by comparing with Wiley9 N11.L Mass Spectral library database.

## RESULTS AND DISCUSSION

### Chemical Analysis by GC-MS

The total detected constituents of pilis oil varied from 29 to 112 components. These were classified into three groups: essential oils, fatty acids and other nonpolar com-

pounds. Not all spectrum peaks of the constituents could be identified due to their low similarity index and the incomplete GC-MS chemical library. Even trace constituents may have functional roles, but the focus of this study was to analyze the main constituents of 16 samples. These main constituents were detected in at least four out of 16 samples (Tables 1 and 2).

### Essential Oils of Pilis

Pilis is made from many plant materials dependant on the producer. The detected essential oils were originally determined from various plant materials used for pilis.<sup>2</sup> The variations in content and composition of essential oils in large jamu factory, local jamu factory, and home based producer were influenced by the kind of material used and production method. Pilis obtained from the large jamu factory and the local jamu factory were preserved in dry packaging. Heating and drying treatment may cause degradation of many constituents, so that there were variations of oil constituents detected in those three pilis producer groups. This drying treatment was aimed to keep pilis for longer period.<sup>5</sup>

Essential oils were very dominant in pilis, especially fresh pilis made by the home-based producers. All Pilis showed the existence of varied essential oils, from one to nineteen main essential oils, although most had 9-19 essential oils (Table 1 and 2). Most of the essential oil components belong to monoterpene group. The number of aromatic essential oils (4 till 9 compounds) was usually less than the nonaromatic one (7 till 12 compounds). Most of the samples had a total amount of aromatic essential oils 2-10 times higher than the nonaromatic oils. The aromatic essential oils detected were estragole, eugenol, methyl salicylate, anethole, zingierone, ethyl methoxycinnamate (p-ethyl-), beta fenchyl alcohol, ar-curcumene, and ar-tumerone. The nonaromatic essential oils were pinene ( $\alpha$ - and  $\beta$ -), caryophyllene, delta cadinene, p-cymene/m-cymene, furanone, gamma terpinene, sabinene, limonene, and cineole. Several essential oils were dominant only in fresh made pilis from home-based producers, namely 4-terpinen-ol, sabinene, estragole, gamma terpinene.

Several essential oil in pilis have specific functions. Anethole has secretolytic, spasmolytic, and carminative effect.<sup>3</sup> Pinene and 1,8-cineol have been reported to have local anesthetic effect.<sup>4</sup> Caryophyllene (isomer  $\alpha$  and  $\beta$ ) has protective effects on the liver (hepatoprotector). Asaron has psychedelic/psychoactive activity, as well as a tranquilizer effect.<sup>4</sup> Eugenol is known for its pain killer, carminative and local anesthetic activities as well as its disinfectant<sup>4</sup> Terpinen-4-ol is useful for relaxation.<sup>6</sup> Beside their posi-

| Table 1. Main Essential Oils of Pilis from Large Jamu Factory and Local Jamu Producer (Relative Amount %) |       |                    |      |      |                     |      |       |       |      |
|---|-------|--------------------|------|------|---------------------|------|-------|-------|------|
| COMPOUND  | CLASS | LARGE JAMU FACTORY |      |      | LOCAL JAMU PRODUCER |      |       |       |      |
|   |       | L JG               | L AM | L MN | O SB                | O GT | O MJ  | O HG  |      |
| Alpha Pinene  | NA    | MH                 | n.d  | 1,08 | n.d                 | n.d  | 0,36  | n.d   | n.d  |
| Beta Pinene   | NA    | SH                 | n.d  | n.d  | n.d                 | 0,18 | n.d   | n.d   | n.d  |
| Alfa.-Copaene   | NA    | SH                 | n.d  | n.d  | n.d                 | n.d  | 0,12  | n.d   | n.d  |
| Alpha humulene  | NA    | SH                 | n.d  | 0,06 | n.d                 | n.d  | n.d   | n.d   | n.d  |
| Alpha-cubebene  | NA    | SH                 | 0,25 | n.d  | n.d                 | 0,45 | 0,6   | n.d   | n.d  |
| Beta selinene   | NA    | SH                 | 0,13 | n.d  | n.d                 | n.d  | 0,17  | n.d   | n.d  |
| Cadina-1,4-diene  | NA    | SH                 | 0,08 | n.d  | n.d                 | 0,3  | 0,6   | 1,05  | n.d  |
| Caryophyllene (Trans-)  | NA    | SH                 | 0,14 | 0,1  | n.d                 | 0,23 | 0,27  | n.d   | n.d  |
| Delta Cadinene  | NA    | SH                 | 0,72 | n.d  | n.d                 | 0,44 | 0,22  | 1,22  | n.d  |
| p-Cymene/<br>m-Cymene   | NA    | MH                 | 3,53 | 0,33 | n.d                 | n.d  | 0,24  | n.d   | n.d  |
| Delta Carene  | NA    | MH                 | n.d  | 0,35 | n.d                 | n.d  | n.d   | n.d   | n.d  |
| Eicosane  | NA    | HC                 | n.d  | n.d  | 1,38                | 0,78 | n.d   | 1,61  | n.d  |
| 2-Furanone  | NA    |                    | 0,21 | n.d  | n.d                 | 1,39 | 5,24  | 2,02  | n.d  |
| Gamma-Terpinene   | NA    | MH                 | n.d  | n.d  | n.d                 | n.d  | n.d   | n.d   | n.d  |
| Sabinene  | NA    | MH                 | n.d  | n.d  | n.d                 | n.d  | n.d   | n.d   | n.d  |
| Limonene  | NA    | MH                 | n.d  | 0,08 | n.d                 | n.d  | 0,06  | n.d   | n.d  |
| Borneol   | NA    | MA                 | n.d  | n.d  | n.d                 | n.d  | 0,21  | n.d   | n.d  |
| 1,8-Cineole   | NA    | MO                 | n.d  | 0,68 | n.d                 | n.d  | 0,17  | n.d   | n.d  |
| Estragole   | AE    | MH                 | n.d  | n.d  | n.d                 | n.d  | n.d   | n.d   | n.d  |
| Methyl salicylate   | AE    | BZ                 | 0,15 | 0,41 | n.d                 | n.d  | 0,22  | 5,66  | 2,48 |
| Anethole  | AE    | MH                 | n.d  | 0,72 | n.d                 | 0,04 | 0,06  | n.d   | n.d  |
| Ar Curcumene  | AE    | SH                 | n.d  | 0,15 | 0,26                | n.d  | n.d   | 0,65  | n.d  |
| Eugenol   | AE    | MH                 | n.d  | 0,72 | 1,27                | n.d  | 7,84  | 13,33 | n.d  |
| Zingiberene   | AE    | SH                 | n.d  | 0,19 | n.d                 | 0,09 | n.d   | n.d   | n.d  |
| Ethyl p-methoxycinnamate  | AE    |                    | 0,74 | 0,37 | n.d                 | n.d  | 2,4   | n.d   | n.d  |
| Beta Fenchyl alcohol  | AE    | MK                 | n.d  | n.d  | n.d                 | n.d  | 0,37  | n.d   | n.d  |
| Ar-tumerone   | AE    |                    | n.d  | n.d  | n.d                 | n.d  | 1,48  | 0,71  | n.d  |
| Asarone (beta)  | AE    | SK                 | n.d  | n.d  | 1,76                | n.d  | 2,99  | n.d   | n.d  |
| Menthol   | AE    |                    | n.d  | n.d  | n.d                 | n.d  | n.d   | 0,65  | n.d  |
| 4-Terpineol   | AE    | MA                 | n.d  | n.d  | n.d                 | n.d  | n.d   | n.d   | n.d  |
| Amount  |       |                    | 5,95 | 5,24 | 2,65                | 3,45 | 20,42 | 26,25 | 2,48 |

**Note :**

L JG : Large Jamu Producer JAGO  
L AM : Large Jamu Producer AIR MANCUR  
L MN : Large Jamu Producer MENEER  
O SB : Local Jamu Producer SAMBETAN  
O GT : Local Jamu Producer GENTONG  
O MJ : Local Jamu Producer MENDJANGAN  
O HG : Local Jamu Producer HANGAT  
n.d : not detected  
AE : Aromatic essential oil  
NA : Non Aromatic essential oil  
MK : Monoterpene keton

MH : Monoterpene hydrocarbon  
SH : Sesquiterpene hydrocarbon  
BZ : Benzenoid  
MA : Monoterpene alcohol  
MO : Monoterpene oxygenated  
HC : Hydrocarbon

Table 2. Main Essential Oils of Pilis from Home Based Producer (Relative Amount %)

| COMPOUND CLASS           | HOME BASED PRODUCER |      |       |       |       |       |      |       |      |       |
|--------------------------|---------------------|------|-------|-------|-------|-------|------|-------|------|-------|
|                          | H KD                | H NB | H IS  | H IR  | H JJ  | H BS  | H SL | H TJ  |      |       |
| Alpha Pinene             | NA                  | MH   | 0,53  | 0,66  | n.d   | 0,46  | 0,13 | 2,69  | n.d  | 0,03  |
| Beta Pinene              | NA                  | SH   | 0,49  | 0,81  | 0,2   | 0,45  | n.d  | n.d   | 0,06 | 0,05  |
| Alfa.-Copaene            | NA                  | SH   | 0,36  | n.d   | 0,06  | 0,07  | n.d  | 0,09  | n.d  | n.d   |
| Alpha humulene           | NA                  | SH   | n.d   | 4,47  | 0,31  | 0,25  | n.d  | n.d   | n.d  | 0,33  |
| Alpha-cubebene           | NA                  | SH   | 0,26  | n.d   | n.d   | n.d   | n.d  | n.d   | n.d  | n.d   |
| Beta selinene            | NA                  | SH   | 0,26  | 0,2   | n.d   | n.d   | n.d  | 1,43  | n.d  | n.d   |
| Cadina-1,4-diene         | NA                  | SH   | 0,06  | n.d   | n.d   | n.d   | n.d  | n.d   | n.d  | 0,33  |
| Caryophyllene (Trans-)   | NA                  | SH   | n.d   | 1,36  | 0,41  | 0,39  | n.d  | 0,65  | 0,05 | 0,37  |
| Delta Cadinene           | NA                  | SH   | 10,94 | n.d   | n.d   | n.d   | n.d  | 0,24  | 0,12 | n.d   |
| p-Cymene/<br>m-Cymene    | NA                  | MH   | n.d   | n.d   | 0,19  | n.d   | n.d  | 0,83  | 0,13 | 0,98  |
| Delta Carene             | NA                  | MH   | 0,98  | n.d   | 0,12  | 0,2   | n.d  | 0,42  | n.d  | n.d   |
| Eicosane                 | NA                  | HC   | n.d   | n.d   | n.d   | n.d   | n.d  | n.d   | 0,24 | 0,15  |
| 2-Furanone               | NA                  |      | n.d   | n.d   | n.d   | n.d   | n.d  | 2,09  | 0,21 | 1,36  |
| Gamma-Terpinene          | NA                  | MH   | 2,25  | n.d   | 0,04  | 0,28  | n.d  | 1,17  | n.d  | 0,03  |
| Sabinene                 | NA                  | MH   | 2,04  | 0,44  | 0,12  | 0,41  | n.d  | 1,48  | n.d  | 0,04  |
| Limonene                 | NA                  | MH   | 0,9   | 0,38  | 0,18  | 0,47  | n.d  | 1,35  | n.d  | n.d   |
| Borneol                  | NA                  | MA   | n.d   | 1,22  | 0,44  | 0,51  | n.d  | 0,32  | n.d  | n.d   |
| 1,8-Cineole              | NA                  | MO   | n.d   | 1,73  | n.d   | 1,25  | n.d  | 2,42  | n.d  | 0,27  |
| Estragole                | AE                  | MH   | 1,17  | 0,7   | 2,78  | 1,64  | n.d  | 0,89  | n.d  | 1,05  |
| Methyl salicylate        | AE                  | BZ   | n.d   | n.d   | n.d   | n.d   | n.d  | n.d   | 0,8  | n.d   |
| Anethole                 | AE                  | MH   | 0,36  | n.d   | 4,57  | 3,2   | n.d  | 1,82  | n.d  | 0,06  |
| Ar Curcumene             | AE                  | SH   | n.d   | n.d   | n.d   | n.d   | n.d  | n.d   | 0,63 | n.d   |
| Eugenol                  | AE                  | MH   | 31,28 | 2,38  | 0,54  | 1,21  | n.d  | 1,46  | n.d  | 10,86 |
| Zingiberene              | AE                  | SH   | 0,08  | n.d   | 0,56  | 1,07  | n.d  | n.d   | n.d  | n.d   |
| Ethyl p-methoxycinnamate | AE                  |      | 10,28 | 0,66  | n.d   | 11,18 | 3,74 | 1,84  | 0,14 | 11,77 |
| Beta Fenchyl alcohol     | AE                  | MK   | 0,33  | 0,25  | n.d   | 0,86  | n.d  | n.d   | n.d  | n.d   |
| Ar-tumerone              | AE                  |      | n.d   | n.d   | n.d   | n.d   | n.d  | 0,33  | 0,43 | 5,74  |
| Asarone (beta)           | AE                  | SK   | n.d   | 0,3   | n.d   | 0,98  | n.d  | n.d   | n.d  | n.d   |
| Menthol                  | AE                  |      | n.d   | n.d   | 0,22  | 0,99  | n.d  | n.d   | n.d  | 0,23  |
| 4-Terpeneol              | AE                  | MA   | 4,94  | 1,43  | 4,54  | 4,00  | n.d  | 7,69  | n.d  | n.d   |
| Amount                   |                     |      | 62,49 | 15,26 | 10,52 | 23,9  | 3,87 | 21,52 | 2,81 | 33,09 |

Note :

H KD : Home Based Producer KENDAL  
H NB : Home Based Producer NYONYA BAHKAN  
H IS : Home Based Producer IYAH SEGAR  
H IR : Home Based Producer IYAH RACIKAN  
H JJ : Home Based Producer JOGJA  
H BS : Home Based Producer BU SARI  
H SL : Home Based Producer SALATIGA  
H TJ : Home Based Producer TANJUNG  
n.d : not detected  
AE : Aromatic essential oil  
NA : Non Aromatic essential oil  
MK : Monoterpene keton

MH : Monoterpene hydrocarbon  
SH : Sesquiterpene hydrocarbon  
BZ : Benzenoid  
MA : Monoterpene alcohol  
MO : Monoterpene oxygenated  
HC : Hydrocarbon

tive effects, several essential oils (e.g. estragole and asaron) were reported to have negative biological activities such as carcinogenic effects.<sup>4</sup> The blending balance of essential oils in pilis have very important role in its therapeutic effects.

Pilis is non invasively administrated. Essential oils of pilis are applied transdermally through direct contact. The absorption of essential oils into the blood stream by dermal contact, has salient pharmacological effects in treating pain, anxiety, sleep disorders, and depression.<sup>7</sup> Topical or transdermal delivery of essential oil from Pilis can penetrate the barrier, the stratum corneum (the outer most layer of skin).<sup>6</sup> Several essential oils, such as 1,8-cineole and menthol can act as natural enhancers because their ability to improve the skin permeation. Moreover, it is reported that menthol and limonene produce maximum permeation of melatonin along with traditional enhancers including fatty alcohols and fatty acids.<sup>1</sup> The combination of two penetration enhancers of different classes such as terpenes (e.g. cineole) and fatty acids (e.g. oleic acid) enhanced transdermal flux of zidovudine in addition to reducing lag time. Some essential oils themselves have been investigated as potential skin penetration enhancers.<sup>6</sup>

### Fatty acids of Pilis

Fatty acids found in significant levels in the pilis from the large jamu factory were the highest, followed by local jamu producer, whilst home-made jamu producer were the lowest. The detected fatty acids were palmitic acid, stearic acid, oleic acids, and linoleic acid. Most of these were present as methyl esters. The role of fatty acids may be important as a carrier or an enhancer. It is not clear whether each producer uses only plant oil materials or adds coconut oil to create a softness to the pilis for more comfortable application.

### Other Nonpolar Components

The other nonpolar constituents detected were dimethoxyphenyl butadiene (DMPBD), shogaol, and members of tetracyclic triterpenes, especially amyryn. DMPBD is the most active anti-inflammatory compound of pilis oil.<sup>12</sup> It was also reported for its antibacterial activity.

Shogaol was reported for antipyretic, analgesic activities, and anti-inflammatory activities such as inhibition of cyclooxygenase-2 (COX-2), to promote digestion activities by peristaltic movement, and central depressant actions such as suppression of motor activity and prolongation of hexobarbital-induced sleep. Shogaol is not found in nature but produced from gingerol due to the loss of water, and probably not found in fresh plant rhizomes, but may result from the extraction process.<sup>4</sup>

Amyryn is a pentacyclic triterpene (PCTs) which has been reported to be an inhibitor of inflammation.<sup>13</sup> The treatment of inflammatory disease can be achieved by giving PCTs. Many PCTs contribute to anti-inflammatory activities.  $\alpha$ ,  $\beta$ -amyryns have shown to exhibit various pharmacological activities *in vitro* and *in vivo* conditions against various health-related conditions, including inflammation, microbial, fungal and viral infections and cancer cells.<sup>8</sup> Triterpenes are promising lead compounds for the development of new multi-targeting bioactive agents.<sup>9</sup> As ingredients of medicinal plants, triterpenes are used in traditional herbal medicine, including the preparation of a semi-solid topical formulation. In particular, lupane-, olean- and ursane triterpenes display various pharmacological effects while being devoid of prominent toxicity. Amyryn is a member of ursane triterpene family. These triterpenes have multi-target properties such as wound healing, anti-inflammatory, anti-bacterial, anti-viral, hepatoprotective and anti-tumoral effects, combined with low toxicity.<sup>9</sup> Oleanolic acid stimulates proliferation of T-cells at lower concentration while betulinic acid and ursolic acid showed inhibitory activity against T-cell proliferation.<sup>11</sup>

### Standard Quality and The Future of Therapeutic Use

The fact that the quality of pilis is diverse denotes that there is no standard quality for the type and quantity of plant materials, the composition of ingredients, as well as the production method of pilis. The quality of herbal medicine is not fixed and rigid. The knowledge and traditional usage are passed from generation to generation orally. The pilis obtained from large jamu producers and local producers were dried. In contrast, pilis from the home based producer was fresh and required no drying treatment. The fresh pilis from home-based producers lasted only several days. The drying treatment of pilis resulted in large losses of essential oils. Therefore, the essential oils were detected in low amounts. Standardization is needed for the efficacy and safety of pilis. Standardization can be based on the right quality and quantity of the plant materials, and standard manufacturing processes. The standard should be determined based on preclinical and clinical experiments. It is necessary to determine the bioactive compounds for standardization of pilis. To date, the commercially available pilis are sold as solid pilis. In the future, a new form of pilis extract may prove to be a better option. The extract or liquid form (distillate) and transdermal patch of pilis could be considered for a modern and more convenient drug delivery.

As a mixture of many plant materials, essential oils and other components have synergetic effects which can immediately penetrate the forehead skin and then diffuse throughout the blood and tissues instantly. Oils cross the



brain-blood barrier and reach the amygdala and other limbic parts of the brain that control our mood, emotions and behavior. They can assist without inducing anxiety, stress, anger or other emotions. The essential oils which contain oxygen molecule(s) can transport nutrient to cells that are nutrient and/or oxygen deprived. Essential oils are often a natural anti-inflammation and are home remedies for pain relief. As a complex mixture of compounds, pilis needs to be further investigated in order to achieve good standard and balance among the individual compounds and at the same time to create a new delivery path as lotion, cream, or transdermal patch. The therapeutic use of pilis is for solving stress or anxiety condition and inflammation either due to the birth process or other related causes.

## CONCLUSION

To our knowledge, this study is the first report on the detection of active compounds, in this case essential oils and other nonpolar compounds from pilis oil. Their main constituents were the essential oils. Some of these have important therapeutic roles. Since the quality of pilis available in the market was varied, it may be preferable to have fresh pilis made by home-producers as reference for creating standards in the future.

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