

# IMPROVEMENT OF SAPONIFICATION EXTRACTION METHOD FOR FATTY ACIDS SEPARATION FROM GEOLOGICAL SAMPLES

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## Introduction

Saponification extraction method has been widely applied for fatty acids separation in the field of organic geochemistry. However, it is proved that this method would lead to great loss of long chain fatty acids and artificial changes of fatty acid compositions. The main reason is the lack of knowledge of the third layer suspends between the aqueous layer and organic layer during the liquid-liquid extraction process. In fact, the third layer consists of lots of long chain carboxylic salts for their special physical and chemical properties. An improved saponification extraction method is proposed and the experimental results show that the carboxylic salts distributed in the third layer could be obtained completely, which greatly enhancing the authenticity and accuracy in experimental analysis of fatty acids and providing new approaches to solve the problem of low extraction efficiency.

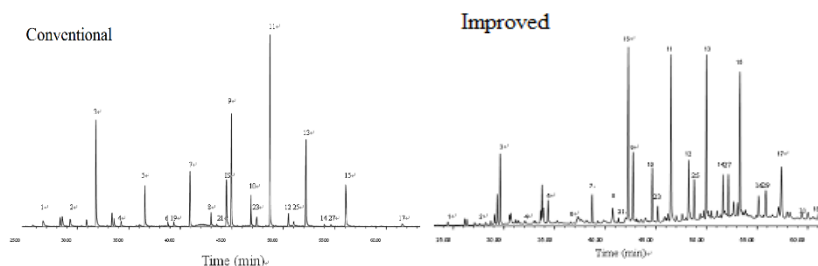
## Methods

The conventional saponification extraction method produces refer to the literature <sup>[1]</sup>.

The peat sample was ground to 200 mesh and extracted in a soxhlet extractor with chloroform for 72h. The extracted material was saponified overnight with 6% KOH-methanol at room temperature. After liquid-liquid extraction with dichloromethane, the aqueous layer and third layer suspends in the middle appeared were added with HCl to pH 1, then fatty acid compounds were obtained with dichloromethane and esterified overnight with 14% boron trifluoride/methanol solution prior to GC/MS analysis and identification.

## Results and Discussion

The reason for the occurrence of the third layer is as follows: (1) long-chain carboxylic salts are weak electrolytes (difficult ionization) and exist still as molecular formations; (2) the physical and chemical properties of carboxylic salts are similar to the anionic surfactants, amphipathic molecules. During the liquid-liquid extraction process, some long chain carboxylic salts would form the third layer with the hydrophilic heads facing to the aqueous layer and the hydrophobic tails inserting into the organic layer<sup>[2]</sup>.



**Figure 1** Total ion current of fatty acids in peat

The comparison of quantitative results is shown in Table 1 and Fig 1. It is shown that the individual fatty acid, especially the medium- to long-chain fatty acids, shows dozens or even hundreds of times of increase compared to that by using the conventional method.

**Table 1** The results contrast of fatty acids obtained by the conventional and improved methods

Fatty acid type	Peak	Carbon number	Conventional method (µg/g)	Improved method (µg/g)	Mass times (Improved /conventional)
Monocarboxylic Acid	1	C <sub>14</sub>	2.0	5.3	2.7
	2	C <sub>15</sub>	0.2	0.8	5.2
	3	C <sub>16</sub>	15.2	149.0	9.8
	4	C <sub>17</sub>	0.0	0.8	25.2
	5	C <sub>18</sub>	16.8	117.2	7.0
	6	C <sub>19</sub>	N.A.	0.7	0.7
	7	C <sub>20</sub>	1.3	17.6	13.1
	8	C <sub>21</sub>	0.1	7.2	139.6
	9	C <sub>22</sub>	1.6	21.3	13.7
	10	C <sub>23</sub>	0.0	13.9	401.6
	11	C <sub>24</sub>	1.9	96.0	51.7
	12	C <sub>25</sub>	0.0	11.7	474.8
	13	C <sub>26</sub>	1.5	177.3	121.5
	14	C <sub>27</sub>	0.0	11.6	740.4
	15	C <sub>28</sub>	1.3	119.3	94.4
	16	C <sub>29</sub>	N.A.	14.0	14.0
	17	C <sub>30</sub>	N.A.	54.2	54.2
	18	C <sub>31</sub>	N.A.	4.0	4.0
Dicarboxylic Acid	19	C <sub>15</sub>	1.0		1.1
	20	C <sub>16</sub>	0.9	0.9	1.1
	21	C <sub>17</sub>	2.7	3.0	1.1
	22	C <sub>18</sub>	1.7	1.8	1.1
	23	C <sub>19</sub>	2.2	24.2	11.2
	24	C <sub>20</sub>	8.9	85.9	9.7
	25	C <sub>21</sub>	4.6	35.1	7.7
	26	C <sub>22</sub>	15.9	159.8	10.1
	27	C <sub>23</sub>	4.3	36.6	8.4
	28	C <sub>24</sub>	15.1	143.3	9.5
	29	C <sub>25</sub>	3.0	15.5	5.2
	30	C <sub>26</sub>	5.5	52.3	9.5
	31	C <sub>27</sub>	N.A.	4.3	4.3
	32	C <sub>28</sub>	N.A.	12.4	12.4
Total			107.5	1391.0	12.9

## References

- [1] Tommaso T, Igor S, Gustaf H, Oleg D, Peter K, Orjan G (2014). Composition and fate of terrigenous organic matter along the Arctic land–ocean continuum in East Siberia: Insights from biomarkers and carbon isotopes. *Geochim Cosmochim Acta*. 133:235–256
- [2] Rosen MJ (1978) Surfactants and interfacial phenomena. Wiley, New York, pp 615–616