

Odour prediction model using odour activity value from pharmaceutical industry

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Data was collected from three pharmaceutical industries (two Western pharmaceutical industries and one traditional Chinese pharmaceutical industry). Odour concentration was measured by the triangular odour bag method; compounds were quantified by gas chromatography-mass spectrometry. The specific objectives were to determine which compounds contributed most to the overall odour emanating from pharmaceutical industry, and develop equations for predicting odour concentration based on compound odour activity value (OAV). OAV is defined as the concentration of a single compound divided by the odour threshold for that compound. The larger the OAV, the more likely that compound would contribute to the overall odour of a complex odour mixture. According to the OAV and regression analyses, we concluded that acetaldehyde, acetone, ethanol and NH₃ were the most likely contributors to the odour in Western pharmaceutical sites. While for the traditional Chinese pharmaceutical site, acetaldehyde, acetone, H₂S, methanal and ethanol were the most likely contributors to the overall odor. Acetaldehyde and Acetone were the compounds with the highest OAV from both Western and Chinese pharmaceutical industries. The multivariate regression analyse results showed that individual OAV was a good predictor of odour concentration for traditional Chinese pharmaceutical industry, the R² of the regression equations ranged from 0.85 to 0.93. While for Western pharmaceutical industry, the odour concentration predictions was poor with R² ranged from 0.30 to 0.65.

1. Introduction

Environmental odors are inherent parts of most industrial sites and may be the cause of an array of reactions, frequently becoming a cause of public environmental discomfort (Carmo, 2010). Offensive odors are not only a direct threat for human health and welfare, but also represent a significant contribution to photochemical smog formation and particulate secondary contaminant emission (Belgiorno et al., 2012). In recent years, much attention was paid on waste disposal facilities such as sewage treatment plant, composting plant; landfill and so on, as well as animal feeding operation plants. Pharmaceutical industry is also an important type of odour pollution source which often causes complaints by surrounding residents. But there is little study about odour characteristics from pharmaceutical industry.

There are hundreds of odorous compounds emitted from pharmaceutical industry. However, it is unlikely that each of these compounds contributes equally to the aroma of a complex odour mixture. For environment management, it is vital to determine which compounds are most responsible for an odor. One of the methods proposed for assessing the relative importance of an individual compound in a complex odour mixture is the odour activity value (OAV). The OAV is defined as the ratio of the concentration of a single compound to the odour threshold for that compound (Friedrich

and Acree, 1998; Trabue et al., 2006). The idea of numerically adding individual OAV to assess overall odour potential was initially proposed by Guadagni (1963) and later by Leffingwell and Leffingwell (1991). When studying combinations of odorants, Audouin et al. (2001) found that OAV provided a poor estimate of odour at higher intensities but was better at lower intensities. Scientists in the food and beverage industries have used OAV to assess odorants. For example, OAV has been used to determine the most important aroma contributors to meat (Grosh, 1994), coffee (Semmelroch and Grosh, 1996), white wine (Guth, 1997), cheese (Qian and Reineccius, 2003), orange juice (Plotto et al., 2004), bread (Hansen and Schieberle, 2005), beer (Fritsch and Schieberle, 2005).

Despite the extensive use of OAV in the study of food and beverages, there has been limited use of OAV in assessment of odorants associated with pharmaceutical field. Conceptually, the larger the OAV, the more likely that compound will contribute to the overall odour of a complex odour mixture. In this research, three pharmaceutical industries were selected as the research objects based on the analyses of the complaint case in Tianjin. We used OAV and multivariate regression techniques for prediction of odors from pharmaceutical industries.

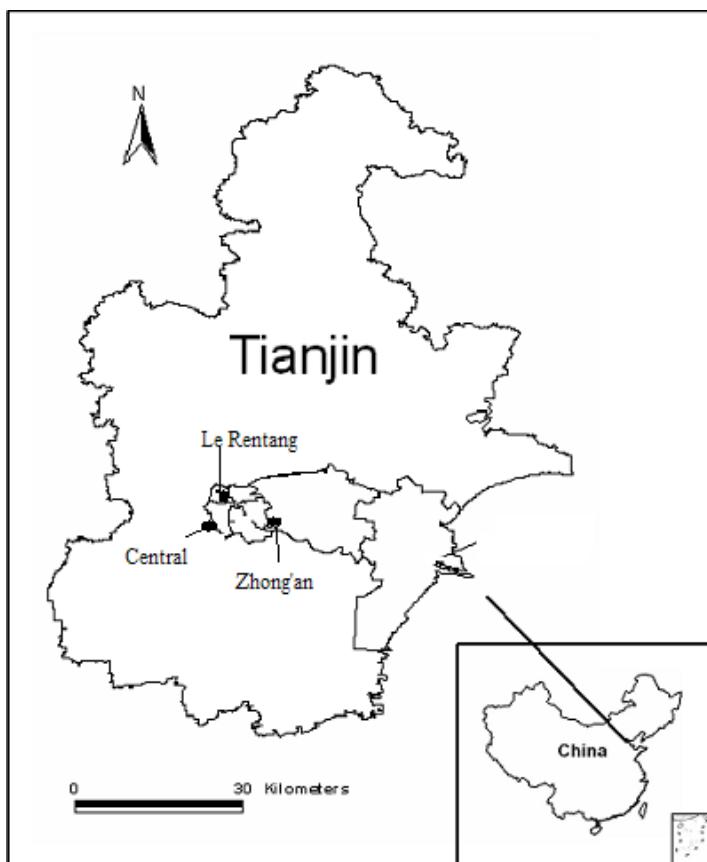


Figure 1: Localization of pharmaceutical sites in Tianjin, China.

The objectives of this study were to (1) analyze the main odorous pollutants at three pharmaceutical sites, (2) find the most significant odorants that contribute to odour concentrations of the site, (3) develop models for predicting odour concentration of pharmaceutical industries using multilinear regressions analyses compound OAV.

2. Materials and methods

2.1 Sampling sites

The study was carried out at three pharmaceutical sites located in Tianjin, China (as shown in Fig. 1). The trials took place in two different periods of the year, summer (2014) and autumn (2014), in order to guarantee the accuracy of the observation results by taking account of different meteorological conditions. Three pharmaceutical industries were selected. The sampling points were shown in Tab. 1.

Table 1: Sampling points

ID	Western pharmaceutical industry	Location	Traditional Chinese medicine industry
P ₁	first production workshops	first extract workshop	
P ₂	second production workshops	second extract workshop	
P ₃	raw material storage	preparation workshop	
P ₄	first synthetic workshop	packing workshop	
P ₅	second synthetic workshop	exhaust funnel	
P ₆	iron sludge treatment	herb residue treatment	
P ₇	wastewater treatment	wastewater treatment	

Note: For Western pharmaceutical industry sampling points, P₁, P₂, P₃ were sampled in Zhong'an pharmaceutical industry, P₄-P₇ were sampled in the central pharmaceutical industry. For traditional Chinese pharmaceutical industry, p₁-p₇ were sampled in Le Rentang pharmaceutical industry.

2.2 Experimental method

Odour concentration was measured by the triangular odour bag method. Compounds were quantified by gas chromatography-mass spectrometry. The quantitative analysis of the sample was according to EPA TO-15 method. Ammonia concentration analysis method referenced Ambient air and exhaust gas-Determination of ammonia-Nessler's reagent spetcrophotomet (HJ 533-2009). Odour analysis method based on Air quality-Determination of odor-Triangle odour bag method (GB/T14675-93).

2.3 Odour activity values

A comprehensive literature review of odour detection thresholds is presented by van Gemert (2003). The single-compound odour threshold (SCOT) is defined as the lowest concentration of a single compound in air that can be detected by the human olfactory sense when compared to a non-odorous sample (Parker et al., 2010). The concentration of the compound can be tested by gas chromatography and other analytical instruments, odour threshold can be obtained by database. Using the concentration of VOC in the air samples from three pharmaceutical industries, OAV were calculated for each individual compound. The geometric mean SCOT value was used for the calculation of OAV (eq.1):

$$OAV = \frac{C}{SCOT} \quad (1)$$

Where OAV is the odour activity value for an individual compound (dimensionless), C is the concentration of the compound ($\mu\text{g m}^{-3}$), and SCOT is the odour detection threshold for the individual compound ($\mu\text{g m}^{-3}$).

2.4 Statistical analysis

Japanese researchers believed that compared with the odour concentration, odour index can reflect the human olfactory sensation better (Iwasaki et al., 1978). Odour concentration and odour index of the sample are calculated by eq. 2:

$$N = 10 \cdot \log OC \quad (2)$$

Where OC=odour concentration, N=odour index.

Multilinear regressions between odour and individual gas OAV were investigated using eq. 3 (SPSS, 2008):

$$N = A_0 + A_1(OAV_1) + A_2(OAV_2) + \dots + A_n(OAV_n) \quad (3)$$

Where OAV₁ through OAV_n are the calculated OAVs of the n individual compounds, A₀, A₁ ... A_n are regression coefficients (i.e., weights applied to the OAV values) determined in the multilinear regression analyses.

According to eq. 1 and eq. 2, prediction equations were also developed using multilinear regression techniques (eq. 4)

$$\log OC = B_0 + B_1(OAV_1) + B_2(OAV_2) + \dots + B_n(OAV_n) \quad (4)$$

Where OC is odour concentration, B₀, B₁ ... B_n are regression coefficients. "Backward method" were used for these analyses. The so-called "backward method" was used for these analyses. This is the most commonly used method. In the backward method, SPSS enters all independent variables into the model. Then the independent variable with the largest p-value ($p > 0.1$) is removed, and the regression is re-calculated. If this weakens the model significantly, the variable is re-entered; otherwise it is deleted. This procedure is repeated until only significant variables remain in the model.

The statistical analyses were also conducted using the MaxR (maximum R² improvement) selection method in SAS version 9.2 (SAS Institute, Inc., Cary, N.C.). The MaxR selection method considers all possible variable combinations to find the best (i.e., the highest R² per the MaxR selection method) one-variable model, the best two-variable model, the best three-variable model, and so on.

3. Results and discussion

3.1 Analyze the the main odorous compounds

For the Zhong'an pharmaceutical industry, the main odorous pollutants were inorganic gas (3.7020 mg/m³) and organic compounds including alkane (0.3395 mg/m³), alkene (0.0215 mg/m³), halohydrocarbon (16.0765 mg/m³), arene (0.1055 mg/m³) and oxygen-containing hydrocarbon (45.3246 mg/m³). The vast majority was oxygen-containing hydrocarbon accounting for 69.12% of the total mass concentration. There were total 40 substances quantitatively detected, containing 8 alkanes, 3 alkene, 8 arene, 10 halohydrocarbon, 9 oxygen-containing hydrocarbon and 2 inorganic gas.

For the central pharmaceutical industry, the total detection concentration was higher in summer (66.5434 mg/m³) than that in autumn (143.3594 mg/m³). The main odorous pollutants were inorganic gas (0.4569 mg/m³) and organic inorganic compounds including alkane (3.8728 mg/m³), alkene (0.6250 mg/m³), arene (51.5933 mg/m³), halohydrocarbon (0.1773 mg/m³) and oxygen-containing hydrocarbon (153.1777

mg/m^3). Oxygen-containing hydrocarbon was also the vast majority accounting for 73% of the total mass concentration. There were total 55 substances quantitatively detected, including 17 alkane, 4 alkene, 14 arene, 8 halohydrocarbon, 11 oxygen-containing hydrocarbon and ammonia.

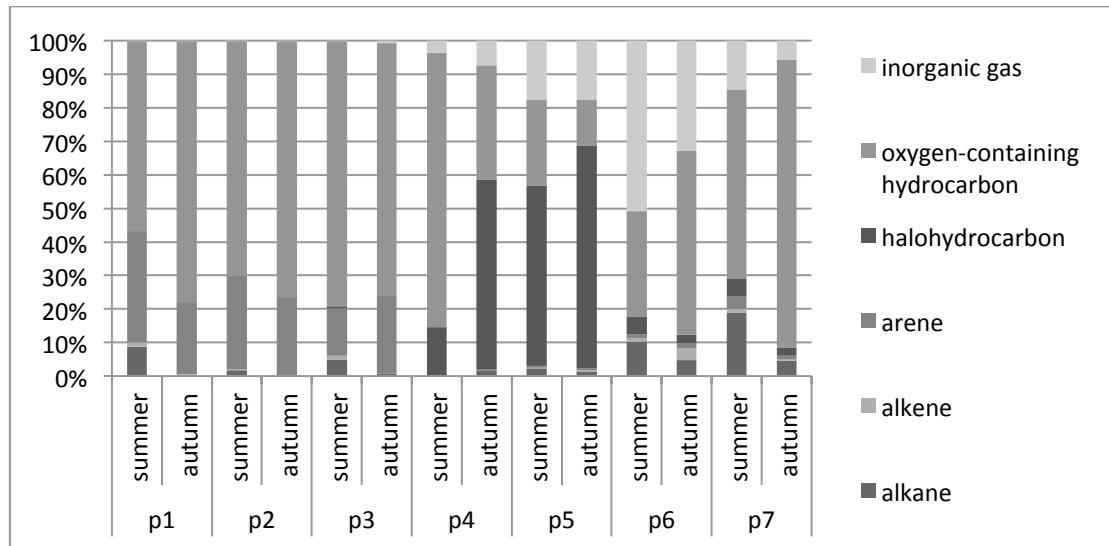


Figure 2: The proportion of pollutants concentration in Western pharmaceutical industry during summer and autumn.

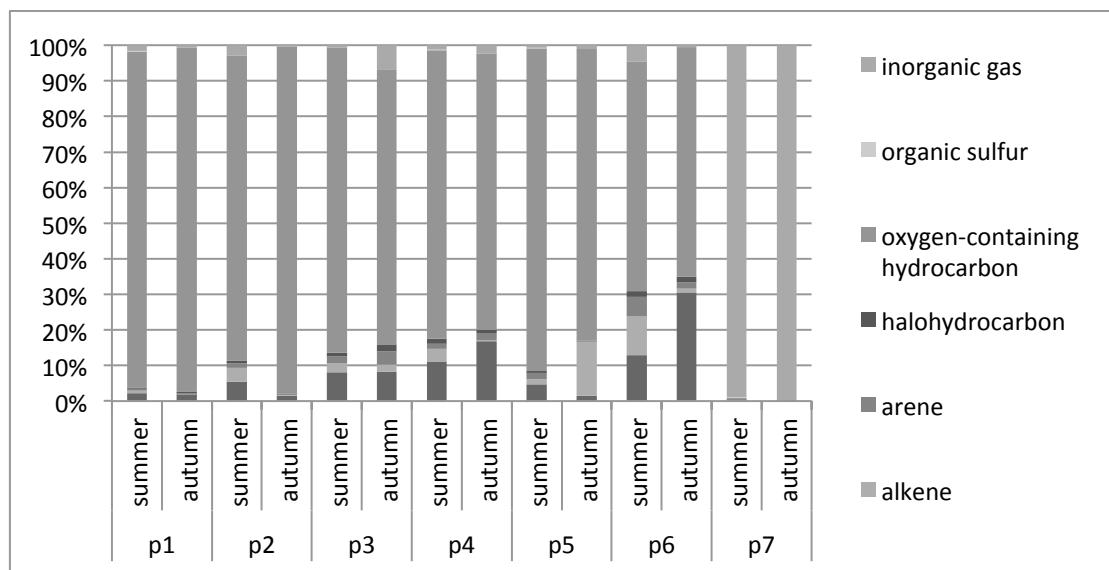


Figure 3: The proportion of pollutants concentration in Western pharmaceutical industry during summer and autumn.

Seven compounds were detected as the major contributor to total detection concentration in Le Rentang pharmaceutical industry, a maximum of 93% of the total mass concentration was attributed to oxygen-containing hydrocarbon compounds ($87.1791 \text{ mg}/\text{m}^3$), 4% to alkane ($3.6131 \text{ mg}/\text{m}^3$), 1% to alkene ($0.7834 \text{ mg}/\text{m}^3$), arene ($0.7455 \text{ mg}/\text{m}^3$) and inorganic gas ($0.9255 \text{ mg}/\text{m}^3$), respectively, the proportion of halohydrocarbon and organic sulfur was slight. There were total 70 substances

quantitatively detected, including 17 alkane, 9 alkene, 10 arene, 17 halohydrocarbon, 14 oxygen-containing hydrocarbon, 2 inorganic gas and 1 organic sulfur.

Fig. 2 and Fig. 3 showed the proportion of pollutants concentration during summer and autumn in Western pharmaceutical industry and traditional Chinese pharmaceutical industry, respectively. It shown that the vast majority was oxygen-containing hydrocarbon compounds in both Western and traditional Chinese pharmaceutical industry. While for P₇ in traditional Chinese pharmaceutical industry, inorganic gas accounting for about 99% of the total mass concentration, it mainly due to hydrogen sulfide was the most significant compounds in wastewater treatment.

Table 2: The top three OAVs of the compounds in each site.

Western medicine industry				traditional Chinese medicine industry			
		summer compound ^[a]	OAV			summer compound	OAV
		compound	OAV			compound	OAV
P	1	Acet	261.31	m-xy	18.57	Acet	610.98
P	2	Ace	153.59	Ace	17.39	Ace	58.29
P	3	m-xy	18.46	Acet	17.10	H ₂ S	26.69
	Odour		41687		309		741
	1	Acet	170.28	Acet	177.75	Acet	624.15
P	2	Ace	143.99	Buta	95.95	Prop	110.61
P	3	m-xy	20.07	Acetone	62.07	H ₂ S	53.67
	Odour		13183		741		2344
	1	Acet	54.30	Acet	221.84	Acet	193.84
P	2	Prop	49.60	Ace	63.21	Ace	62.99
P	3	Acet	44.06	m-xy	4.59	Etha	3.07
	Odour		41687		417		Meth
	1	Prop	14765	Acet	37.57	Acet	193.84
P	2	Ace	50.04	Ace	8.11	H ₂ S	26.41
P	3	Acet	36.68	H ₂ S	6.51	Ace	3.93
	Odour		132		98		H ₂ S
	1	Ace	229.22	Acet	35.81	Acet	1459
P	2	Acet	41.67	Isov	31.01	Acet	154.75
P	3	NH ₃	0.48	Acet	22.84	H ₂ S	25.37
	Odour		234		174		Ace
	1	Acet	42.17	Acet	29.63	Acet	1318
P	2	Ace	28.52	Ace	17.44	H ₂ S	234
P	3	NH ₃	0.73	NH ₃	0.36	Ace	54.49
	Odour		977		74		Meth
	1	Acet	42.84	Acet	79.93	H ₂ S	550
P	2	Ace	21.95	H ₂ S	31.18	Acet	174.68
P	3	NH ₃	0.09	Ace	1.75	Ace	79.68
	Odour		4169		417		Ace
	1	Acet	42.84	Acet	79.93	H ₂ S	36.02
P	2	Ace	21.95	H ₂ S	31.18	Acet	108.87
P	3	NH ₃	0.09	Ace	1.75	Ace	36.34
	Odour		4169		417		Ace

^[a] Acet = acetaldehyde, Ace = acetone, m-xy = m-xylene, H₂S = hydrogen sulfide, Buta = butanone, Prop = propanal, Isov = isovaleral, Etha=ethanol, Meth = meyhanal, NH₃ = ammonia.

3.2 Single-compound odour activity value

The larger the OAV, the more likely that compound would contribute to the overall odour of a complex odour mixture. In order to compare the sensory stimulation strength of single-compound OAV and analyse their contribution, the top three OAV of the compounds were provided in Tab. 2.

The main odorous pollutants in Western pharmaceutical industry were inorganic gas and oxygen-containing hydrocarbon compounds. The compounds with highest frequency at each sampling point were acetaldehyde, acetone, followed by ethanol, ammonia. While the OAV value of ethanol and ammonia were lower, therefore, acetaldehyde and acetone were found as the most significant compounds in Western pharmaceutical industry.

For traditional Chinese pharmaceutical industry, the main odorous pollutants were inorganic gas, oxygen-containing hydrocarbon compounds, and less alkene and arene compounds. Acetaldehyde, acetone, hydrogen sulfide were found as the most significant compounds in this sampling point due to their highest frequency.

Odour concentration was found to be large difference in two seasons, the values in summer were larger than that in autumn, this was expected, odour and gas concentrations and emission rates were significantly different due to variations in the sampling point and management characteristics of the sites. It may be also due to the samples was interfered by other undetected gas compounds in summer.

3.3 Single-compound odour activity value

The multivariate regression analyses for the Western medicine industry and traditional Chinese medicine industry sites yielded numerous multi-parameter prediction models for odour concentration.

The OAV was not a good predictor of odour concentration in Western pharmaceutical industry, that is one-parameter model (Acetaldehyde only, $R^2=0.30$) to a 4-parameter model with $R^2=0.65$ (Tab. 3). The most significant compounds were Acetaldehyde, Acetone, Ethanol and NH_3 . These particular compounds apparently can be used to account for up to 65% of the variance in odour concentrations. There was no serious collinearity among the independent variables. The linear regression equation was as follows:

$$\log OC = 27.262 + 0.008X_1 + 0.023X_2 + 1.652X_3 - 5.465X_4 \quad R^2=0.65, P<0.05 \quad (5)$$

Where OC was the predicted odour concentration; X_1 , X_2 , X_3 , X_4 were the OAV of Acetaldehyde, Acetone, Ethanol, NH_3 , respectively. P-value($p<0.05$) suggested the equation had a good statistical significance.

While for traditional Chinese medicine industry site, the multilinear regression results ranged from a best one-parameter model (Acetaldehyde only, $R^2=0.85$) to a 5-parameter model with maximum $R^2=0.93$ (Table 4). The most significant compounds were Acetaldehyde, Acetone, Methanol, Ethanol and H_2S . These particular compounds apparently can be used to account for up to 93% of the variance in odour concentrations. The collinearity was good among the independent variables. The linear regression equation is as follows:

$$\log OC = 29.867 + 0.007x_1 - 0.068x_2 - 8.994x_3 - 0.214x_4 - 0.001x \quad R^2=0.93, P<0.05 \quad (6)$$

Where OC was the predicted odour concentration; x_1 , x_2 , x_3 , x_4 , x_5 were the OAV of Acetaldehyde, Acetone, Methanol, Ethanol, H_2S , respectively. P-value($p<0.05$) suggested the equation had a good statistical significance.

According to these results, all correlations were statistically significant ($p<0.05$), but a maximum of 65% of the variation in odour concentrations could be predicted by using

OAV in Western medicine industry, and a maximum of 93% of the variation in traditional Chinese medicine industry. It was concluded that OAV can be used to predict odour concentrations from Pharmaceutical Industry, but these OAV will not always yield high coefficients of determination. It was mainly because odour was sampled in different site and the effect of seasonal changes on odour and gas concentrations.

Table 3: Regression coefficients and corresponding R^2 values for the model in eq. 5 (Western medicine industry site). Shown are coefficients for n = 1 to 4 parameter models.

No. of parameters	Intercept	Compound				R^2	P
		Acetaldehyde	Acetone	Ethanol	NH_3		
1	24.255	0.058	-	-	-	0.30	0.12
2	23.654	0.051	0.020	-	-	0.34	0.21
3	24.396	0.021	0.018	1.866	-	0.56	0.03
4	27.262	0.008	0.023	1.652	-5.465	0.65	0.03

Based on OAV analysis and the regression analyses, we noticed that Acetaldehyde and Acetone had the highest frequency and their OAV were higher, there was no doubt that they were the highest contributors to odour in pharmaceutical industry.

Table 4: Regression coefficients and corresponding R^2 values for the model in equation 6 (traditional Chinese medicine industry site). Shown are coefficients for n = 1 to 5 parameter models.

No. of parameter s	Intercept	Compound						R^2	P
		Acetaldehyde	Acetone	Methanol	Ethanol	H_2S			
1	29.250	-4.55	-	-	-	-	-	0.85	0.31
2	29.311	0.001	-0.013	-	-	-	-	0.85	0.61
3	31.555	-0.029	-0.002	-11.162	-	-	-	0.92	0.00
4	36.870	0.008	-0.142	-19.315	-0.643	-	-	0.93	0.00
5	29.867	0.007	-0.068	-8.994	-0.214	-0.001	-	0.93	0.00

4. Conclusions

The following conclusions were drawn from this research:

From analyzing the total detection concentration, it shown that oxygen-containing hydrocarbon compounds had the largest proportion in both Western medicine industries and traditional Chinese medicine industry.

When odour activity values were taken into account, the most significant compounds were propanal acetaldehyde and acetone for Western pharmaceutical industry, acetaldehyde, acetone, isovaleral and hydrogen sulphide for traditional Chinese pharmaceutical industry. Both Western pharmaceutical and traditional Chinese pharmaceutical industry had the same two compounds with the highest OAVs (ranked high to low: acetaldehyde, acetone).

Although the odour concentration predictions was generally poor ($R^2=0.30$ to 0.65) in Western pharmaceutical industry, individual OAVs was a good predictor of odour concentration using multivariate regression analyses for traditional Chinese pharmaceutical industry ($R^2=0.85$ to 0.93).

Based on the OAV and regression analyses, we concluded that acetaldehyde, acetone, ethanol and NH₃ were the most likely contributors to the Western pharmaceutical sites. While for the traditional Chinese pharmaceutical sites, acetaldehyde, acetone, H₂S, methanal and ethanol were the most likely contributors.

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