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Sulfite Management during Vinification and Impact on the Flavor of Solaris Wine

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Abstract: Effective sulfur dioxide (SO_2) management is crucial in winemaking to minimize oxidative changes in wine flavor during storage. This study explored the impact of various SO_2 management techniques on Solaris white wine's flavor components and sensory properties. Five treatments were administered: 'SO_2 in juice' (50 mg/L SO_2 added to juice pre-fermentation), 'Control' (60 mg/L SO_2 added post-fermentation), 'Low SO_2' (50 mg/L SO_2 post-fermentation), 'High SO_2' (100 mg/L SO_2 post-fermentation), and 'No SO_2' (no SO_2 added). The 'Control' followed a standard procedure, in which the achieved level of free sulfite is measured and extra SO_2 added to reach the recommended level of free sulfite for the pH of the wine. Here, 50 + 10 mg/L was added. Volatile compounds were analyzed using dynamic headspace sampling coupled with gas chromatography–mass spectrometry after 0, 3, 6, and 12 months of storage. Sensory evaluation by a trained panel after 12 months revealed stronger perceptions of 'overall impression', 'chemical', 'bitter', 'overripe fruit', and 'honey' notes in the 'No SO_2' and 'SO_2 in juice' wines. The data underscore the significant influence of SO_2 management on the flavor stability of Solaris white wines, emphasizing the need for strategic SO_2 interventions during winemaking to enhance sensory quality over time.

Keywords: sulfur dioxide management; oxidation; white wine; sensory evaluation; acetaldehyde; volatile compounds

Citation: Toldam-Andersen, T.B.; Zhang, S.; Liu, J.; Bredie, W.L.P.; Petersen, M.A. Sulfite Management during Vinification and Impact on the Flavor of Solaris Wine. *Fermentation* **2024**, *10*, 210. https://doi.org/10.3390/fermentation10040210

Academic Editor: Maurizio Ciani

Received: 27 February 2024
Revised: 22 March 2024
Accepted: 10 April 2024
Published: 12 April 2024

1. Introduction

During the storage of white wines, there is a potential for the loss of initial freshness and fruity properties. Setting aside issues caused by various microorganisms, the primary concern for the degradation of young wine quality lies in chemical changes, especially alterations in the aroma compound profile due to diverse types of reactions. Notably, oxidation (of ethanol), Strecker, and early-stage Maillard reactions are considered significant contributors to flavor changes during the storage of white wine [1].

Free SO_2 stands out as the most commonly used preservative agent in wine. The addition of SO_2 is often conducted already to the must, where it serves to prevent must oxidation and inhibit the growth of undesirable microorganisms during fermentation [2]. In bottled wine, it not only limits acetaldehyde formation but also binds acetaldehyde, thereby protecting or enhancing the wine's aroma. Extensive research has been conducted on the preservative function of SO_2 on wine volatile compounds. For instance, Jackowetz et al. [3] found that the level of SO_2 significantly affects acetaldehyde production and degradation during alcoholic fermentation. Similarly, Garde-Cerdán and Ancín-Azpilicueta [4] explored wine stored with SO_2 in bottles, revealing higher concentrations of volatile compounds, particularly esters and alcohols, compared to wine aged in bottles without SO_2. SO_2 also influences carbonyl aging-related compounds in red wines [5], where the increase

in oxidative compounds may be a consequence of aldehydes forming bisulfites once SO_2 undergoes oxidation. However, few studies have specifically addressed the effects of SO_2 levels on acetaldehyde and other oxidation-related compounds during the aging of white wine [3,4]. Specifically, wines with a high content of acetaldehyde may require more SO_2 to attain adequate levels of free or active SO_2. In this context, the timing of SO_2 addition is crucial in winemaking. The amount added is also pivotal and depends especially on pH, as well as on wine style and cultivar [2]. White wines are often intended for consumption within a year after release. Adding low levels of SO_2 may fail to protect the wine from early oxidation, while high levels may pose a risk of inducing health problems in sensitive consumers and have adverse effects on sensory quality. Therefore, there is an evident need for a more profound understanding of the role SO_2 plays in relation to wine aging, particularly concerning the development of volatile compounds.

In order to evaluate if changes in volatile compounds are large enough to actually impact the sensory quality of wine, sensory evaluation is needed. Descriptive sensory methods are frequently employed in characterizing wine flavor [6,7]. These methods rely on samples that represent a relatively extensive sensory space to obtain clear discrimination. However, when sample variations are subtle, perceiving minor differences with these methods can be challenging. Instead, the difference from the control test emerges as a practical and sensitive method for use in food quality control programs, such as those for wine [8] and cheese [9].

Solaris is the primary cultivar grown in Denmark for white wine production. This interspecific hybrid cultivar holds high value for organic wine production in cool and cold climate regions due to its excellent disease tolerance and early ripening properties. As the wine industry expands in Nordic countries, there is a growing need to understand how to control wine quality from these cold climate cultivars. In a previous study, we assessed the quality of a selection of Danish Solaris wines and observed that differences in vintage seemed less characteristic than differences arising from sulfur management by producers [10]. The wines were segregated into two main clusters: half were associated with fruity and floral descriptors, while the other half was characterized by less pleasant flavors. Combining data on free and total SO_2 and vinification methods by producers, oxidation appeared to result from poor sulfite management. The present work aims to investigate the effect of storage time and SO_2 addition practice (timing and amount) on the profiles of volatile compounds and their relationship to the sensory properties of Solaris white wines.

2. Materials and Methods

2.1. Chemical Standards

Chemical standards for volatile compounds were sourced from reputable suppliers: Sigma-Aldrich (St. Louis, MO, USA), Fluka (Madrid, Spain), and Aldrich (Madrid, Spain). Ethanol (HPLC grade, 99.9%) and L (+)-tartaric acid (>99.5%) were procured from Sigma-Aldrich (Kiev, Ukraine).

2.2. Winemaking

Grapes of the Solaris cultivar were hand harvested in Pometet, Copenhagen University, Denmark, at a sugar content of 22.6 °Brix, with total acidity (as tartaric acid) at 10.2 g/L and pH at 3.03. The grapes were destemmed, crushed, and directly pressed in a 40 L hydropress (Speidel). As depicted in Figure 1, five treatments involving various SO_2 management strategies were conducted in duplicate. In the first treatment, 50 mg/L of SO_2 (sulfur dioxide water solution at 5% *m/v*) was added to the juice for SO_2 stabilization two days before fermentation (SO_2 in juice). During this period, all batches were placed in a cold room (3 °C) for clearing. The clear juice was racked into 5 L fermenters filled with CO_2 prior to racking to minimize oxidation. Each treatment is in duplicate. In the remaining four treatments, SO_2 was administered post-fermentation. The treatment receiving an addition of 60 mg/L SO_2 was labeled as the 'Control.' It followed a standard procedure involving

pH-dependent SO_2 addition, a common practice in wine production [11]. Initially, 50 mg of SO_2 was added (similar to the 'Low SO_2' treatment); however, after assessing the free sulfite level and pH, an extra 10 mg of SO_2/l was added to achieve the recommended SO_2 level at the wine's current pH, equating to a molecular SO_2 level of 0.8 ppm. Samples with added SO_2 concentrations of 0, 50, and 100 mg/L were designated as 'No SO_2,' 'Low SO_2,' and 'High SO_2' wines, respectively. All fermentations were conducted in a 5 L bioreactor maintained at 18 °C in a temperature-controlled environment. To initiate fermentation, 0.2 g/L of commercial yeast *Saccharomyces cerevisiae*, *bayanus* (Lalvin DV10™ from Lallemand, Fredericia, Denmark) was added to all samples. Throughout fermentation, samples were collected from the musts on a daily basis for density and acetaldehyde analysis until all wines reached dryness. Density was measured with a DMA35 Density meter from Anton Paar (Tokyo, Japan). Following fermentation, the wines were carefully racked, and varying amounts of SO_2 were added (see Figure 1). SO_2 addition occurred shortly after fermentation, when acetaldehyde concentrations were at their lowest, aiming to minimize SO_2 binding and optimize free SO_2 levels. Free and total SO_2 levels were measured immediately post-bottling. All wines were bottled in 375 mL bottles with screw caps and stored in a wine cellar under dark conditions at a temperature range of 15–18 °C. Chemical analyses were conducted periodically during storage (at 0, 3, 6, and 12 months post-bottling), with sensory evaluations performed on the wines after 12 months of storage.

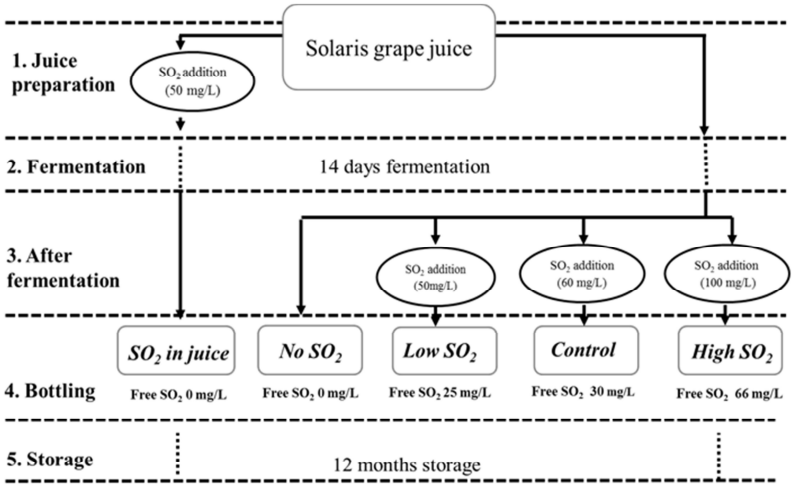

Figure 1. The experimental design and sample names were used for each SO_2 management.

2.3. SO_2 Measurement

The measurement of free and total sulfur dioxide (SO_2) utilized a modified Ripper iodine redox titration method, following the protocol outlined by Tanner and Sandoz [12]. Total and free SO_2 levels in the wine were assessed at 3, 6, and 12 months post-bottling.

2.4. Analysis of Volatile Compounds

Volatile compound analysis followed a previously published method [6]. Dynamic headspace sampling (DHS) was employed to extract aroma compounds. Each wine sample (20 mL) was transferred to a 100 mL flask, to which 1 mL of 4-methyl-1-pentanol solution in water (5 mg/L, Aldrich, Steinheim, Germany) was added as an internal standard. Volatile compounds were trapped on a Tenax-TA trap (200 mg, mesh size 60/80, Buchem BV, Apeldoorn, The Netherlands) using a purge volume of 2 L (100 mL/min for 20 min). The trapped volatile compounds were then analyzed using a thermal desorption gas chromatography mass spectrometry system (Perkin Elmer TurboMatrix 350, Shelton, CT, USA, coupled to a 7890A GC/5975C VL MSD from Agilent Technologies, Palo Alto, CA, USA), equipped with a DB-Wax column (Agilent J&W, Palo Alto, CA, USA, 30 m × 0.25 mm × 0.25 mm). Data were analyzed using MSD Chemstation G1701EA software

(Version E.01.00.237, Agilent Technologies Inc., Palo Alto, CA, USA), with compound identification based on mass spectra comparison with a standard library (Wiley275.l, HP product no. G1035A). Linear retention indices (RI) were calculated using a homologous series of alkanes (C5–C22) for further verification of compound identification. The RI values were compared to the RI of authentic standards or reported literature RI. Quantification of volatile compounds relied on calibration curves established using synthetic wine, as detailed in previous work [6]. Duplicate analyses were performed on each biological duplicate. Acetaldehyde quantification employed a modified DHS method described by Zhang and Petersen [13], taking acetaldehyde's high volatility and low breakthrough volume in Tenax-TA traps into account. The method included conditions similar to those described above, except that headspace purge volume was decreased to 80 mL (40 mL/min for 2 min) and a lower cryo-focusing temperature (−20 °C) was used in the second step of the thermal desorption.

2.5. Sensory Analysis

A sensory evaluation of the finished wines after 12 months of storage was conducted using the difference from the control method [14,15] in standard sensory booths at the University of Copenhagen. An external panel with nine trained panelists (seven female and two male) participated and conducted the testing with informed consent.

2.5.1. Panel Training

Three training sessions, each lasting 1 ½ h, were conducted. During the initial session, five pairs of samples (sample names as indicated in Figure 1) and a list of wine fault attributes were presented to the panelists. Each pair contained two samples: a blind sample of the four treatments and the control sample (SO_2 addition of 60 mg/L), which was used as a reference. The fifth pair was control vs. control. In each pair, the panelists were asked to evaluate differences between the control and test wines' overall impressions using an open evaluation sheet and checking wine fault attributes from the list provided. This led to a total of 13 descriptive terms. Subsequent sessions introduced reference standards for each term, and then the wines were tasted again, allowing panelists to mark the degree of difference between paired samples. In discussion with the panel, the eight most relevant attributes were selected. In the final training session, panelists evaluated four pairs of samples to familiarize themselves with the evaluation procedure and the difference from control scaling.

2.5.2. Panel Wine Evaluation

Approximately 20 mL of wine, served at 9 °C, was presented in ISO standard wine glasses with watch-glass lids. Cold water and crackers were provided for palate cleansing. Samples were presented in pairs, with the control wine and an experimental wine coded with three-digit numbers. Panelists were instructed to taste the wine pairs and evaluate the difference in pre-determined sensory attributes between each sample and the control using a 15 cm unstructured line scale with anchors labeled "no difference" and "extreme difference." Sample pairs were presented randomly to each assessor and evaluated in one session, with sessions repeated three times on different days.

2.6. Data Analysis

A one-way analysis of variance (ANOVA) was conducted on the concentrations of volatile compounds using SPSS (IBM SPSS Statistics v.22, Chicago, IL, USA), with 'sample' treated as a fixed effect. For sensory data analysis, a two-way ANOVA was employed, considering 'product' as a fixed factor and 'assessor' as a random factor. Tukey's post hoc test was subsequently utilized to ascertain the extent of differences between samples, with a significance level set at 5%.

3. Results and Discussion

3.1. Acetaldehyde Production and Degradation during Wine Fermentation

Acetaldehyde stands out as the most significant carbonyl compound quantitatively produced by yeast during alcoholic fermentation. Figure 2A illustrates that, excluding the 'SO_2 in juice' sample, acetaldehyde production peaked (400–500 mg/L) around fermentation days 2–3 for all samples, followed by a rapid decline over the subsequent three days, in line with density changes (Figure 2B). While no significant differences in peak acetaldehyde values were observed between the control and other treatments, the 'SO_2 in juice' sample displayed a lower peak acetaldehyde level than the control. The delayed onset of fermentation, documented by the delayed decrease in density of the 'SO_2 in juice' treatment, illustrates the antimicrobial impact of sulfite addition even on a strong cultural yeast [2]. The impact of SO_2 addition on acetaldehyde production can be due to its influence on various pathways. These are, for example, the alcohol dehydrogenase-catalyzed formation of acetaldehyde from ethanol by mitochondria in yeast [16] and the pyruvate decarboxylase-catalyzed decarboxylation of pyruvate, which has acetaldehyde as the end product [17]. The bonding of acetaldehyde with SO_2 could also provide an explanation. However, the final acetaldehyde concentrations were comparable across all samples. The patterns shown in Figure 2 are very similar to those found by other authors [3].

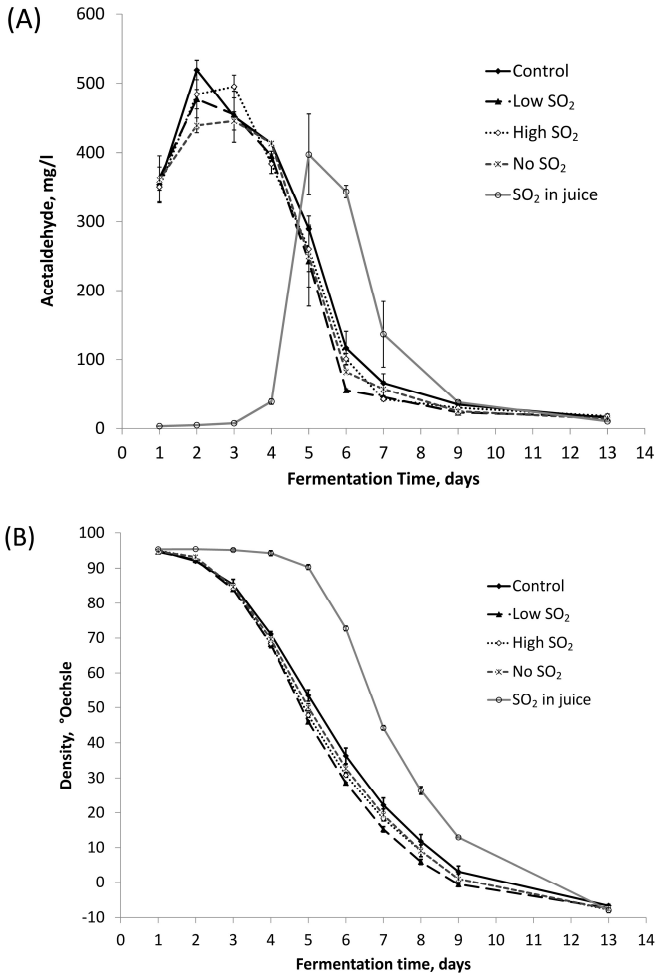

Figure 2. The production and degradation of (**A**) acetaldehyde and (**B**) change in density during fermentation. The values shown are averages of fermentation replicates with a standard deviation.

3.2. Free SO_2 Levels and Acetaldehyde Concentration during Wine Storage

The consumption of SO_2 serves as a reliable indicator of wine oxidation. In our study, significant decreases in SO_2 levels were observed during bottle aging, particularly within

the first 6 months (Figure 3B). The 'Low SO_2' sample experienced the most significant relative loss of free SO_2 (87.1% after 12 months). Although the wine with high SO_2 addition retained a level of approximately 24.5 mg/L after 12 months, expected to safeguard the wines at a pH of approximately 3.1 [18], the remaining samples fell below 10 mg/L after 12 months, a level potentially concerning for continued wine aging.

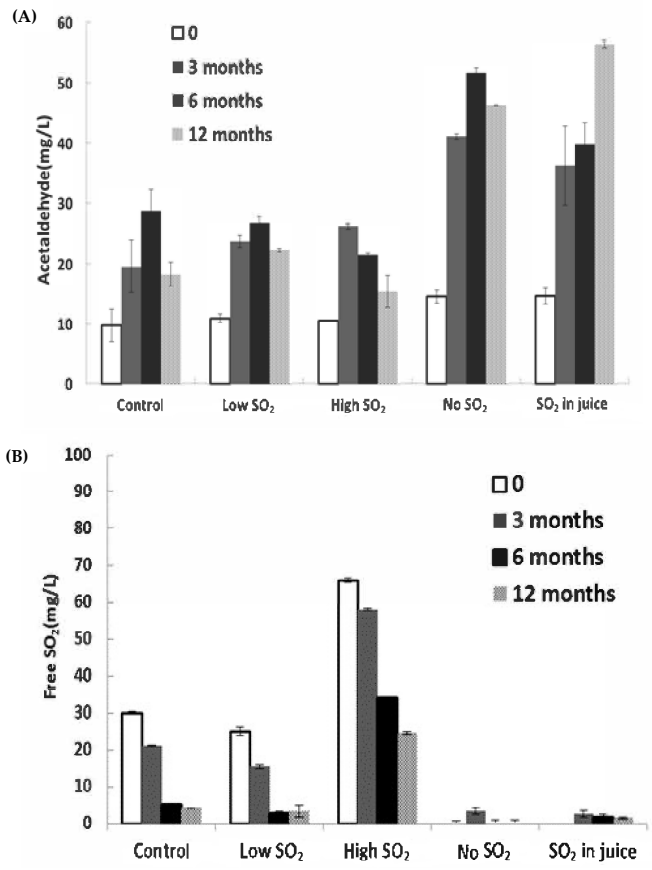

Figure 3. Concentrations of (**A**) acetaldehyde and (**B**) free SO_2 in wines during storage. The values shown are averages of fermentation replicates with a standard deviation.

Acetaldehyde can also arise post-alcoholic fermentation through ethanol oxidation when exposed to air [19]. Acetaldehyde concentrations did not significantly differ among wines immediately after fermentation. However, post-bottling, acetaldehyde markedly increased until 6 months of aging in all wines, particularly in the 'No SO_2' and 'SO_2 in juice' samples, which contained the lowest free SO_2 levels (Figure 3A,B). After 12 months of aging, significant differences were observed between the control wine and the wine without SO_2 addition. The lowest acetaldehyde level was found in the 'High SO_2' sample, although not significantly different from the control wine. SO_2 can inhibit aldehyde formation by competing with hydrogen peroxide, which induces ethanol oxidation [3]. The decline in acetaldehyde over 12 months of storage could be attributed to SO_2 depletion, aligning with the findings of Bueno et al. [20]. Alternatively, it could result from rapid polymerization reactions of wine phenolics mediated by acetaldehyde [21,22], or acetaldehyde may decrease through reactions with alcohols to form dioxolanes [23].

3.3. Changes in Volatile Compounds during 3, 6, and 12 Months of Storage

Volatile compound variations were monitored during storage at 3, 6, and 12 months post-bottling (Table 1). Several acetate esters, such as propyl acetate, 2-methylpropyl acetate, hexyl acetate, and phenethyl acetate, declined during storage across all wine samples. These esters often create fruity aromas like banana and pears and floral aromas like rose. Conversely, several ethyl esters, including ethyl 2-methylbutyrate, ethyl 3-methylbutyrate,

ethyl pyruvate, and ethyl 9-decenoate, increased over time. This is in accordance with the general observation that many acetate esters decrease during bottle storage while ethanol directly reacts with organic acid to generate a range of ethyl esters [24].

Table 1. Changes in volatile compounds during storage at 3, 6, and 12 months (μg/L). Averages of all treatments. Only compounds with a *p*-value < 0.001 are shown.

Compounds	3 Months	6 Months	12 Months	*p*-Value
Propyl acetate	226	200	87	<0.001
2-Methylpropyl acetate	83	63	20	<0.001
Butyl acetate	13.0	7.1	2.3	<0.001
Hexyl acetate	760	627	149	<0.001
Phenethyl acetate	1270	446	185	<0.001
Ethyl 2-methylbutyrate	0.9	4.3	6.2	<0.001
Ethyl 3-methylbutyrate	3.9	17.1	21.6	<0.001
Ethyl pyruvate	97	231	362	<0.001
Methyl octanoate	1.8	2.3	3.9	<0.001
Ethyl 9-decenoate	8.0	26.0	46.7	<0.001
Diethyl succinate	208	2400	7750	<0.001
(E)-2-Hexen-1-ol	2.3	5.7	13.5	<0.001
(Z)-2-Hexen-1-ol	10.8	13.6	17.0	<0.001
1-Heptanol	10.6	23.6	39.1	<0.001
1-Octanol	3.8	5.5	7.7	<0.001
Linalool	69	92	129	<0.001
Hotrienol	45	59	84	<0.001
Furfural	4.3	13.2	53.7	<0.001
Neroloxide	11.2	32.7	49.0	<0.001
Vitispirane	1.7	11.1	13.0	<0.001
2,4,5-Trimethyl-1,3-dioxolane	0.0	690	910	<0.001

Additionally, certain compounds known as markers for oxidation, including β-damascenone and vitispirane isomers, demonstrated substantial changes during storage. β-Damascenone, usually imparting fruity and floral notes, exhibited a slight decrease after 12 months of bottle aging, in line with the findings of Chisholm et al. [25] on aged Vidal blanc wine. Conversely, mono-terpene alcohols like linalool and hotrienol increased in wines without SO_2 protection during storage, consistent with the observations of Zoecklein et al. [26] in aged Riesling white wines.

Monoterpenes can undergo considerable fluctuations due to isomerization and/or breakdown, potentially influenced by biochemical rearrangement in addition to hydrolysis [27]. During storage, significant increases were observed in the levels of 2,4,5-trimethyl-1,3-dioxolane (associated with green and phenolic notes), furfural (bread, almond, and sweet aromas), diethyl succinate, and ethyl pyruvate (with fruity, sweet, vegetable, and caramel nuances) in the 'No SO_2' wine. Previous studies by others have also noted similar rises in these compounds in oxidized or aged wines [28–30]. Escudero et al. [28] identified 2,4,5-trimethyl-1,3-dioxolane as a key odorant in oxidized wine. Therefore, these compounds may serve as valuable indicators of wine aging.

The increase in 2,4,5-trimethyl-1,3-dioxolane levels is attributed to oxygen exposure, as it undergoes a condensation reaction with 2,3-butanediol and acetaldehyde [23]. Consequently, the rise in acetaldehyde concentration during wine aging coincides with the formation of 2,4,5-trimethyl-1,3-dioxolane. Similar findings were reported in beer by Vanderhaegen et al. [31], supporting this observation.

Among the compounds mentioned in this section, however, only ethyl 2-methylbutyrate, ethyl 3-methylbutyrate, linalool, and 2,4,5-trimethyl-1,3-dioxolane are present in concentrations above the threshold (Table 2). These compounds are further discussed in the next section.

Table 2. Volatile compounds were quantified in the wines with different SO_2 managements. Values are presented as averaged concentrations over replicates (μg/L) in finished wines after 12 months of storage. Levels not connected by the same letter are significantly different, and significance levels are presented as 'ns' ($p > 0.05$), '*' ($p \leq 0.05$), '**' ($p \leq 0.01$), or '***' ($p \leq 0.001$).

Compounds	Odor Threshold [1]	Calculated LRI [2]	Standard LRI [3]	Odor Description [4]	Concentrations of Wines					Sig.	Log OAV [5]				
					Control	Low SO_2	High SO_2	No SO_2	SO_2 in Juice		Control	Low SO_2	High SO_2	No SO_2	SO_2 in Juice
Esters															
Ethyl propanoate	1800 (1)	971	962	Fruit	360	340	330	330	340	ns	−0.7	−0.7	−0.7	−0.7	−0.7
Ethyl 2-methylpropanoate	**15 (2)**	**969**	**969**	**Sweet, rubber**	**51**	**50**	**51**	**46**	**48**	**ns**	**0.5**	**0.5**	**0.5**	**0.5**	**0.5**
Ethyl butanoate	**20 (2)**	**1038**	**1040**	**Apple**	**320**	**290**	**310**	**290**	**310**	**ns**	**1.2**	**1.2**	**1.2**	**1.2**	**1.2**
Ethyl 2-methylbutyrate	**1 (2)**	**1053**	**1058**	**Apple**	**7.1 a**	**5.8 b**	**6.4 ab**	**5.4 b**	**6.4 ab**	*	**0.9**	**0.8**	**0.8**	**0.7**	**0.8**
Ethyl 3-methylbutyrate	**3 (2)**	**1072**	**1079**	**Fruit**	**24 a**	**19 b**	**21 ab**	**19 b**	**24 a**	*	**0.9**	**0.8**	**0.8**	**0.8**	**0.9**
Ethyl pentanoate	94 (3)	1153	1150	Yeast, fruit	0.89	0.73	0.79	0.68	0.70	ns	−2.0	−2.1	−2.1	−2.1	−2.1
Ethyl 2-butenoate	-	1178	1174	-	6.9	6.8	7.5	6.7	7.1	ns					
Ethyl hexanoate	**5 (2)**	**1263**	**1255**	**Apple peel, fruit**	**940**	**880**	**900**	**850**	**880**	**ns**	**2.3**	**2.2**	**2.3**	**2.2**	**2.2**
Ethyl pyruvate	100,000 (4)	1284	1286	Ethereal, fruity, sweet, vegetable, caramel	170 cd	200 c	110 d	790 a	540 b	***	−2.8	−2.7	−3.0	−2.1	−2.3
Ethyl (*E*)-3-hexenoate	-	1327	1327	Pineapple, fruity	0.32 a	0.28 ab	0.24 bc	0.23 bc	0.22 c	**					
Ethyl heptanoate	220 (1)	1354	1351	Fruit	1.1 a	0.91 ab	0.80 b	0.76 b	0.54 c	***	−2.3	−2.4	−2.4	−2.5	−2.6
Ethyl lactate	157,360 (1)	1353	1353	Fruit	96 c	100 bc	110 ab	97 bc	110 a	*	−3.2	−3.2	−3.2	−3.2	−3.2
Ethyl octanoate	**14 (5)**	**1447**	**1450**	**Fruit, fat**	**2200**	**2060**	**2100**	**2000**	**2100**	**ns**	**2.2**	**2.2**	**2.2**	**2.2**	**2.2**
Ethyl nonanoate	377 (6)	1548	1553	Fruity, rose, waxy, Tropical	2.8	2.0	2.3	1.8	2.4	ns	−2.1	−2.3	−2.2	−2.3	−2.2
Ethyl furoate	16,000 (7)	1641	-	-	17 b	15 b	10 c	23 a	22 a	***	−3.0	−3.0	−3.2	−2.8	−2.9
Ethyl decanoate	**200 (7)**	**1649**	**1651**	**Grape**	**930**	**870**	**880**	**830**	**890**	**ns**	**0.7**	**0.6**	**0.6**	**0.6**	**0.6**
Diethyl succinate	200,000 (1)	1691	1689	Wine, fruit	7700	7400	8700	6400	8600	ns	−1.4	−1.4	−1.4	−1.5	−1.4
Ethyl 9-decenoate	100 (8)	1703	1705	Fruit	53	54	47	42	37	ns	−0.3	−0.3	−0.3	−0.4	−0.4
Ethyl laurate	500 (9)	1854	1861	Leaf	160	124	150	120	150	ns	−0.5	−0.6	−0.5	−0.6	−0.5
Propyl acetate	4700 (1)	981	978	Sweet, fruity	96 a	91 ab	83 b	81 b	81 b	*	−1.7	−1.7	−1.8	−1.8	−1.8
2-Methylpropyl acetate	1600 (10)	1017	1018	Fruit, apple, banana	19	20	20	19	19	ns	−1.9	−1.9	−1.9	−1.9	−1.9
Butyl acetate	1880 (1)	1078	1082	Pear	2.1	2.2	2.2	2.1	2.6	ns	−3.0	−2.9	−2.9	−3.0	−2.9
3-Methylbutyl acetate	**30 (2)**	**1140**	**1142**	**Banana**	**2100**	**2060**	**2100**	**2100**	**2200**	**ns**	**1.8**	**1.8**	**1.8**	**1.8**	**1.9**
Hexyl acetate	1500 (1)	1299	1293	Fruit, herb	150	150	150	150	140	ns	−1.0	−1.0	−1.0	−1.0	−1.0
(*Z*)-3-Hexenyl acetate	-	1327	1328	Green, banana	0.21	0.20	0.18	0.18	0.18	ns					
(*E*)-3-Hexenyl acetate	-	1333	1337	Sweet, green, sharp-fruity	3.3	3.7	3.7	3.1	3.7	ns					
Heptyl acetate	-	1385	1386	-	2.7 a	2.6 a	2.1 ab	2.0 b	1.2 c	**					
Phenethyl acetate	250 (2)	1837	1835	Rose, honey, tobacco	160 b	170 b	190 b	160 b	240 a	***	−0.2	−0.2	−0.1	−0.2	0.0
Methyl hexanoate	84 (11)	1198	1196	Fruit, fresh, sweet	1.0	0.89	0.96	0.85	0.98	ns	−1.9	−2.0	−1.9	−2.0	−1.9
Methyl octanoate	-	1400	1401	Orange	4.2	3.8	3.9	3.3	4.1	ns					
3-Methylbutyl octanoate	125 (7)	1668	1672	-	27	23	24	21	27	ns	−0.7	−0.7	−0.7	−0.8	−0.7
Alcohols															
1-Propanol	9000 (12)	1041	1041	Alcohol, pungent	82 c	130 ab	110 abc	140 a	100 bc	**	−2.0	−1.8	−1.9	−1.8	−2.0
2-Methyl-1-propanol	40,000 (2)	1104	1100	Wine, solvent, bitter	570	1200	890	1100	620	ns	−1.8	−1.5	−1.7	−1.6	−1.8
1-Butanol	150,000 (5)	1164	1165	Medicine, fruit	210 b	270 a	260 a	270 a	280 a	*	−2.9	−2.7	−2.8	−2.7	−2.7
3-Methyl-1-butanol	**30,000 (2)**	**1237**	**1238**	**Whiskey, malt, burnt**	**131,000 a**	**132,000 a**	**126,000 b**	**130,000 a**	**132,000 a**	*	**0.6**	**0.6**	**0.6**	**0.6**	**0.6**
1-Pentanol	64,000 (1)	1279	1274	Balsamic	60 a	48 b	46 b	44 b	60 a	***	−3.0	−3.1	−3.1	−3.2	−3.0
2-Heptanol	200 (8)	1341	1340		3.0	2.7	2.8	2.5	2.9	ns	−1.8	−1.9	−1.9	−1.9	−1.8
Hexanol	8000 (2)	1373	1372	Resin, flower, green	2700 a	2500 b	2400 b	2500 b	2200 c	***	−0.5	−0.5	−0.5	−0.5	−0.6

Table 2. *Cont.*

Compounds	Odor Threshold [1]	Calculated LRI [2]	Standard LRI [3]	Odor Description [4]	Concentrations of Wines					Sig.	Log OAV [5]				
					Control	Low SO_2	High SO_2	No SO_2	SO_2 in Juice		Control	Low SO_2	High SO_2	No SO_2	SO_2 in Juice
(*E*)-3-Hexenol	150,000 (1)	1382	1386	Grass	25 ^{b}	23 ^{b}	25 ^{b}	23 ^{b}	31 ^{a}	***	−3.8	−3.8	−3.8	−3.8	−3.7
3-Ethoxy-1-propanol	-	1390	1370	-	25 ^{a}	16 ^{b}	19 ^{b}	17 ^{b}	18 ^{b}	***					
(*Z*)-3-Hexenol	400 (2)	1398	1390	Grass	34 ^{b}	34 ^{b}	34 ^{b}	35 ^{b}	47 ^{a}	***	−1.1	−1.1	−1.1	−1.1	−0.9
(*E*)-2-Hexen-1-ol	15,000 (13)	1421	1420	Green, leaf, walnut	13	13	12	15	15	ns	−3.1	−3.1	−3.1	−3.0	−3.0
(*Z*)- 2-Hexen-1-ol	-	1430	1430	Leaf, green, wine, fruit	18	16	18	16	17	ns					
1-Heptanol	-	1468	1471	Chemical, green	44	42	37	34	38	ns					
2-Ethyl-hexanol	8000 (8)	1502	1499	Rose, green	1.0 ^{a}	0.89 ^{ab}	0.89 ^{ab}	0.71 ^{c}	0.75 ^{bc}	*	−3.9	−4.0	−4.0	−4.1	−4.0
1-Octanol	900 (8)	1570	1573	Chemical, metal, burnt	9.1 ^{a}	7.5 ^{b}	7.7 ^{b}	7.1 ^{b}	7.0 ^{b}	*	−2.0	−2.1	−2.1	−2.1	−2.1
Benzyl alcohol	200,000 (5)	1896	1897	Sweet, flower	1.1	1.2	1.2	1.0	1.2	ns	−5.3	−5.2	−5.2	−5.3	−5.2
2-Phenylethanol	**10,000 (2)**	**1936**	**1935**	**Honey, spice, rose, lilac**	**13,200 ^{b}**	**9600 ^{cd}**	**12,800 ^{bc}**	**9500 ^{d}**	**16,900 ^{a}**	**	**0.1**	**0.0**	**0.1**	**0.0**	**0.2**
Aldehydes															
Acetaldehyde	**500 (2)**	**701**	**701**	**Pungent, overripe fruit**	**18,400 ^{d}**	**22,400 ^{c}**	**15,500 ^{e}**	**46,400 ^{b}**	**56,000 ^{a}**	***	**1.6**	**1.7**	**1.5**	**2.0**	**2.0**
3-Methylbutanal	**4.6 (1)**	**921**	**917**	**Malty**	**3.0 ^{c}**	**3.9 ^{b}**	**2.3 ^{c}**	**6.8 ^{a}**	**6.8 ^{a}**	***	**−0.2**	**−0.1**	**−0.3**	**0.2**	**0.2**
Hexanal	9.1 (14)	1087	1087	Grass, tallow, fat	1.5 ^{bc}	2.4 ^{b}	0.57 ^{c}	4.6 ^{a}	4.7 ^{a}	***	−0.8	−0.6	−1.2	−0.3	−0.3
Octanal	-	1313	1311	Fat, soap, lemon, green	0.82	0.91	0.77	0.97	0.81	ns					
Nonanal	15 (5)	1405	1402	Fat, citrus, green	1.6 ^{a}	1.2 ^{ab}	0.68 ^{b}	1.6 ^{a}	1.5 ^{a}	*	−1.0	−1.1	−1.3	−1.0	−1.0
2-Furfural	14,100 (15)	1464	1461	Bread, almond, sweet	53 ^{b}	50 ^{b}	13 ^{c}	80 ^{a}	72 ^{a}	***	−2.4	−2.5	−3.0	−2.2	−2.3
Decanal	10 (5)	1510	1511	Soap, orange peel, tallow	0.51 ^{bc}	0.63 ^{b}	0.39 ^{c}	0.89 ^{a}	1.1 ^{a}	***	−1.3	−1.2	−1.4	−1.1	−1.0
Benzaldehyde	2000 (12)	1541	1537	Almond, burnt sugar	3.2	4.7	1.9	3.0	3.9	ns	−2.8	−2.6	−3.0	−2.8	−2.7
Ketones															
2-Heptanone	-	1192	1190	Soap	1.5 ^{a}	1.1 ^{c}	1.1 ^{c}	1.2 ^{bc}	1.4 ^{ab}	*					
6-Methyl-5-hepten-2-one	-	1352	1365	Mushroom, earthy	0.28	0.22	0.23	0.24	0.3	ns					
2-Nonanone	-	1386	1388	Hot milk, soap, green	0.76 ^{a}	0.46 ^{b}	0.55 ^{b}	0.52 ^{b}	0.54 ^{b}	*					
Terpene and C-13 norisoprenoids															
Limonene	15 (5)	1200	1200	Lemon, orange	0.33 ^{ab}	0.37 ^{a}	0.28 ^{c}	0.36 ^{a}	0.30 ^{bc}	**	−1.7	−1.6	−1.7	−1.6	−1.7
Neroloxide	-	1482	1485	Floral	54	49	47	42	52	ns					
Vitispirane	800	1547	1533	Floral, woody	15	13	10	12	15	ns	−1.7	−1.8	−1.9	−1.8	−1.7
Linalool	**15 (10)**	**1559**	**1560**	**Flower, lavender**	**140 ^{a}**	**130 ^{a}**	**82 ^{b}**	**140 ^{a}**	**150 ^{a}**	***	**1.0**	**0.9**	**0.7**	**1.0**	**1.0**
β-Damascenone	**0.05 (2)**	**1841**	**1844**	**Apple, rose, honey**	**4.3 ^{b}**	**4.2 ^{b}**	**0.98 ^{c}**	**6.5 ^{a}**	**5.9 ^{a}**	***	**1.9**	**1.9**	**1.3**	**2.1**	**2.1**
Hotrienol	100 (14)	1623	1621	Floral	90 ^{a}	86 ^{a}	54 ^{b}	91 ^{a}	99 ^{a}	***	0.0	−0.1	−0.3	0.0	0.0
α-Terpineol	250 (10)	1712	1716	Oil, anise, mint	13	12	12	12	13	ns	−1.3	−1.3	−1.3	−1.3	−1.3
Other compounds															
2,4,5-trimethyl-1,3-dioxolane	**900 (16)**	**941**	**940**	**Green, phenolic**	**310 ^{c}**	**310 ^{c}**	**23 ^{d}**	**2100 ^{a}**	**1800 ^{b}**	***	**−0.5**	**−0.5**	**−1.6**	**0.4**	**0.3**
Dihydro-2-methyl-3(2*H*)-thiophenone	-	1543	1518	-	0.34 ^{b}	0.44 ^{b}	0.40 ^{b}	0.27 ^{b}	1.0 ^{a}	***					

[1] Odor threshold in references (1) [32] The matrix was a 12% water/ethanol mixture. (2) [33] The matrix was an 8.10 g/100 g water/ethanol solution. (4) [30] The matrix was 14% (*v*/*v*) ethanol solutions. (3), (11) and (14) [34–36] The matrix was water. (5), (8) [37,38] The matrix was a 10% water/ethanol solution containing 5 g/L of tartaric acid at pH 3.2. (6) [39] threshold in the 11% ethanol solution. (7), (12), (13), and (15) [40–43] The matrix was an 8.91 g/100 g water/ethanol solution containing 7 g/L glycerol and 5 g/L tartaric acid, with the pH value adjusted to 3.4 with 1 M NaOH; (9) [44] The matrix was a 14% water/ethanol solution adjusted to pH 3.5 with tartaric acid. (10) [45] The matrix was a 9.72 g/100 g water/ethanol solution containing 5 g/L of tartaric acid at pH 3.2. (16) [23]. [2] The retention indices (RIs) of volatiles were calculated as the retention time of the volatiles normalized to the retention times of adjacently eluting n-alkanes (C6–C22). [3] Linear retention indices (LRI) were calculated from authentic standard compounds analyzed on the same system. [4] Odor descriptions based on flavornet.org (accessed on 12 February 2024) and www.thegoodscentscompany.com (accessed on 12 February 2024) online databases, except 2,4,5-trimethyl-1,3-dioxolane whose odour descriptors are from [23] and [28]. [5] OAV: Odor activity value was calculated by dividing the concentration by the odor threshold value of the compound. Compounds with a log OAV > 0 in at least one sample are in bold.

3.4. Effect of SO_2 Management on Volatile Compounds in Final Wines (12 Months of Storage)

Table 2 presents the identified and quantified volatile compounds in the finished wine (12 months post-bottling). A total of 68 volatile compounds, including 31 esters, 17 alcohols, 8 aldehydes, 7 terpenes, 3 ketones, 1 sulfur compound, and a dioxolane, were identified. Given that the contribution of volatile compounds to wine aroma depends on their concentration surpassing the perception threshold, the odor activity value (OAV) was introduced to identify potent odorants. OAV was calculated as the ratio between the compound concentration and its odor threshold. Results revealed that 15 out of 68 quantified volatile compounds exceeded the odor threshold (log OAV > 0) in the wine, suggesting their potential as aroma contributors. Among them, eight esters, notably ethyl hexanoate (apple peel, fruit) and ethyl octanoate (fruit, fat), exhibited high OAV values, contributing fruity and fatty nuances to the wine. The robust presence of esters in Solaris white wines has been previously reported by Liu et al. [10]. While concentrations of these esters did not significantly differ between wines or displayed minor variations, indicating minimal influence from SO_2 treatments, seven volatile compounds with OAV log values above 0 in at least one sample exhibited significant differences between treatments. These included acetaldehyde (pungent, overripe fruit odor), 3-methylbutanal (malty), and 2,4,5-trimethyl-1,3-dioxolane (green, phenolic), with notably higher concentrations in wines with 'No SO_2' and 'SO_2 in juice.' These findings align with previous research demonstrating elevated levels of acetaldehyde and 2,4,5-trimethyl-1,3-dioxolane in oxidized Solaris wines [10]. When sulfur dioxide is added to wine containing free acetaldehyde, it forms a sulfonated adduct [20,46], potentially explaining the low acetaldehyde content in wines with post-fermentation SO_2 addition.

The presence of the acetal 2,4,5-trimethyl-1,3-dioxolane significantly increased in wines exhibiting the highest acetaldehyde levels. Vanderhaegen et al. [47] proposed that an equilibrium between 2,4,5-trimethyl-1,3-dioxolane, acetaldehyde, and 2,3-butanediol could rapidly establish due to the escalating acetaldehyde concentration. This molecule has also been suggested as a marker for detecting oxidation in bottled beer during aging [47].

β-Damascenone, known for its profound impact on wine aroma owing to its low odor threshold value [48], contributes delicate notes of apple, rose, and honey to white wines. In this study, β-Damascenone exhibited the highest concentration in the 'No SO_2' wine, followed by 'SO_2 in juice', and the lowest concentration in the 'High SO_2' wine. This finding is consistent with the model wine study conducted by Daniel et al. [49], which demonstrated that the reaction between sulfur dioxide and β-damascenone yields 4-oxo-4-(2,6,6-trimethylcyclohexa-1,3-dien-1-yl) butane-2-sulfonate as the major non-volatile adduct. Linalool, contributing flowery notes, exhibited variations similar to those of β-damascenone. It is, however, unclear whether similar reactions with sulfur dioxide take place.

3.5. Effect of SO_2 Management on Sensory Properties in Final Wines (12 Months of Storage)

Half of the attributes used in the sensory vocabulary had previously been selected to describe oxidized wines [10,50]. The 'Control' wine served as the sensory reference in the difference from control test, which enables the detection of subtle sensory differences by directly comparing attributes with the control sample in paired evaluations.

ANOVA of the panel difference ratings revealed that all attributes significantly discriminated among wine samples with different SO_2 management (see Table 3). Specifically, the 'No SO_2' and 'SO_2 in juice' wines exhibited significantly stronger flavors compared to the 'Control' and 'High SO_2' wines, except for 'citrus.' It is reasonable to attribute the greater 'chemical', 'honey', and 'overripe fruit' flavors in the 'No SO_2' and 'SO_2 in juice' wines to oxidation. The presence of elevated levels of acetaldehyde in these Solaris wines may contribute to the 'chemical' and 'overripe fruit' flavors. The 'honey' flavor could be attributed to the high levels of phenylethyl acetate and 2-pheneylethanol. Sensory analyses supported the significant impact of the absence of SO_2 during storage on flavor attributes associated with oxidation.

Table 3. Results from the difference from control sensory profiling, rating the intensity differences from the four experimental Solaris wines to the control (addition of 60 mg/L SO_2). All wines were stored for 12 months.

Sensory Attributes	Reference Materials	Low SO_2	High SO_2	No SO_2	SO_2 in Juice	*p*-Value
Overall impression	None	0.7	1.1	6.8 ^{a}	5.2 ^{a}	<0.001
Chemical	2 mL ethanol (99.9%), 200 µL ethyl acetate, 20 µL white vinegar in 25 mL wine	0.8	1	5.8 ^{a}	4.7 ^{a}	<0.001
Citrus	2 mL each of fresh grapefruit and lemon juice and some peel	0.1	0.7	2.5 ^{a}	2.7 ^{a}	<0.001
Bitter	0.015 g quinine sulfate in 1 L water	0.7	1.4	4.3 ^{a}	3.9 ^{a}	<0.001
Flower	12 mL elderflower juice + 1 µL rose flavor	−0.4	0.1	2.3 ^{a}	2.2 ^{a}	<0.001
Honey	3.5 g honey in 25 mL wine	0.1	0.0	1.9 ^{a}	2.1 ^{a}	<0.001
Overripe fruit	12 g overripe apple in 25 mL wine	0.2	0.6	4.6 ^{a}	4.1 ^{a}	<0.001
Lactic acid	16 g buttermilk (Arla, Denmark) in 25 mL wine	−0.3	0.4 ^{a}	3.7 ^{b}	2.2 ^{ab}	<0.001

Note: Control: 60 mg/L of SO_2; Low SO_2: 50 mg/L of SO_2; High SO_2: 100 mg/L of SO_2. Values in a row without superscript letters were not significantly different from the control; samples with different superscript letters were significantly different from each other (Tukey HSD, $p < 0.05$).

No significant sensory differences were perceived between the 'Control' and 'High SO_2' wines, nor between the 'No SO_2' and 'SO_2 in juice' wines. Despite the 'High SO_2' wine containing significantly higher levels of volatile compounds such as nonanal and decanal, which contribute citrus notes, as well as compounds like linalool, β-damascenone, and hotrienol, which impart floral notes, these differences were not detected in the sensory analysis.

4. Conclusions

This study investigated the impact of sulfur dioxide (SO_2) addition on the volatile and sensory characteristics of Solari's white wine. The presence of free SO_2 notably decreased the levels of free acetaldehyde during wine storage while also decreasing the concentrations of other aldehydes, particularly at higher dosage levels. Conversely, in the absence of free SO_2, acetaldehyde levels increased significantly, accompanied by elevated levels of its associated compound, 2,4,5-trimethyl-1,3-dioxolane, carrying 'green' and 'phenolic' aroma notes, both surpassing well above their respective odor thresholds. Additionally, other volatile compounds such as 3-methylbutanal and β-damascenone increased in these wines, likely contributing to amplified sensory impressions of 'chemical', 'overripe fruit', and 'honey' notes.

In the finished wines after 12 months of storage, regardless of SO_2 management, crucial esters defining Solari's wine aroma remained unaltered in concentration. However, even a low addition of sulfite improved the sensory quality significantly. This underscores the significance of employing moderate levels of SO_2 post-Solaris wine fermentation to avoid oxidation of the final product, but higher SO_2 additions conferred no significant benefits at the timespan studied. The progressive loss of free sulfite and, thus, oxidative protection was very evident during storage, and the expected lifespan of the wine needs to be taken into account when the sulfite level is defined pre-bottling.

Author Contributions: Conceptualization, T.B.T.-A. and M.A.P.; methodology, T.B.T.-A., M.A.P. and W.L.P.B.; formal analysis, S.Z., J.L. and M.A.P.; investigation, S.Z.; resources, W.L.P.B., M.A.P. and T.B.T.-A.; data curation, S.Z., M.A.P. and T.B.T.-A.; writing—original draft preparation, S.Z. and J.L.; writing—review and editing, S.Z., J.L., T.B.T.-A., M.A.P. and W.L.P.B.; visualization, S.Z.; supervision, M.A.P., W.L.P.B. and T.B.T.-A.; project administration, T.B.T.-A. and M.A.P.; funding acquisition, M.A.P., W.L.P.B. and T.B.T.-A. All authors have read and agreed to the published version of the manuscript.

Funding: This work was financially supported by The Danish Ministry of Food, Agriculture and Fisheries (GUDP, No 34009-13-0725), Faculty of Science, the University of Copenhagen, and a grant from the Chinese Scholarship Council (No 201206300044).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Data Availability Statement: Raw data can be obtained by contacting M.A.P.

Acknowledgments: We are truly thankful for the technical assistance of the staff at the Pometum and in the labs.

Conflicts of Interest: The authors declare no conflicts of interest. The funders had no role in the design of the study, in the collection, analysis, or interpretation of data, in the writing of the manuscript, or in the decision to publish the results.

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