Sorption of sulfuryl fluoride into wheat and its impact on efficacy, fluoride residues and product quality

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DECLARATION

I certify that the substance of this thesis has not already been submitted for any degree and is not currently being submitted for any other degree or qualification.

I certify that any help received in preparing this thesis, and all sources used, have been acknowledged in this thesis.

[redacted]

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SUMMARY

This work was carried to investigate sorption of sulfuryl fluoride (SF) and its impact on efficacy, residues and technological quality of wheat and its products. An important factor to consider in the practical application of fumigants is the impact of sorption of the gas into the commodity during fumigation. Sorption may reduce the biological activity of a fumigant by reducing the concentration of gas available to target insects and no information is available on the impact of sorption on the biological efficacy of this fumigant against target pests.

Information on the efficacy of SF against insect pests is generally limited to fumigation times of 48 h or less. The aim of this work was to provide more detailed information about the sorption behaviours of SF when wheat, semolina and flour are fumigated at three concentration x time exposures under different conditions (temperature, grain moisture content), filling ratio and repeated fumigation. The impact of fumigating with SF under these conditions on the efficacy against lesser grain borer in the presence of bread wheat, effects on the technological properties of durum wheat and the presence of fluoride residue in selected grain fractions were studied.

For sorption studies, bread wheat, durum wheat, commercial flour and semolina at typical grain storage temperatures (15, 25 and 35°C) and moisture contents (12% and 15%), were fumigated with SF at CT, concentration x time combinations, equal to 1500 mg.h/L (4.167 mg/L x 360 h, 8.928 mg/L x 168 h and 31.25 mg/L x 48 h). The results of this study indicated sorption rate of SF into the commodity increased as temperature and commodity moisture content increased at each applied concentration. The highest rates of sorption occurred at 35°C and 15% m.c., and lowest rates at 15°C and 12% m.c. Importantly, there was no desorption of SF by the commodity after airing under any of the test conditions. My results indicated that SF is sorbed slowly by wheat grains and their processed products (flour or semolina) relative to other common fumigants such as phosphine and methyl bromide.

Sorption follows first order reaction kinetics described by the exponential decay equation, $C_t = C_0\ e^{-k*t}$, where k is the sorption rate constant. Unbound SF is rapidly lost from the commodity upon aeration with no further desorption detected indicating that sorbed SF is irreversibly bound to the commodity matrix. The most important factors determining the rate of sorption are commodity particle size (exposed surfaces) and temperature then moisture. The rapid desorption of SF is beneficial for work place and health and safety. A special consideration needs to be taken into account when fumigation is done with this fumigant in the presence of the commodity to avoid downgrading the toxic level of the fumigant.

Sorption of SF at various grain filling ratios (0.95, 0.75, 0.50) was evaluated with durum wheat fumigated with 1 mg/L SF applied for 168 h at 25°C. The highest sorption rate was at 0.95, and time to sorb the fumigant decreased with increasing filling ratio. Physical sorption accumulated exponentially, and chemical sorption increased linearly. Physical sorption was strongly related to chemical sorption in a quadratic manner, and the independency of physical sorption affected the turning point of the chemical sorption. Sorption of the fumigant increased as the density increased and it was sorbed significantly higher at bulk density than at grain density. this shows the impact of the density of the commodity on sorption as there are more grains in agiven volume with bulk grain. Different densities of the bulk can be obtained from the same grain density and from the same bulk. While it is beneficial to maximise the amount of grain stored in a facility (high filling ratio), this can lead to more extensive sorption of SF and result in higher residue content than using a lower, 0.5 filling ratio. The high sorption of the fumigant at the higher filling ratio left higher fluoride residues as the results of this study indicated. In addition, high sorption means reducing the effective concentration needed to kill eggs and adults therefore downgrading the fumigant concentration under the level required for insect control. It is recommended that a filling ratio of 0.50 be used so that an accurate and effective fumigation procedure against insects with minimal sorbed gas by the grain will be achieved.

Repeated fumigation occurs in the grain industry and this was investigated by testing the impact of up to 5 repeated fumigations of bread (hard) wheat and soft wheat with 8.928 mg/L of SF for 168 h at 25 C. SF was sorbed more into bread wheat than soft wheat. For both commodities, sorption rate decreased with increasing number of fumigations. Fluoride residues increased with increasing number of fumigations, and the maximum residue was at the fifth fumigation. Repeating fumigation four times or more results in the fluoride residue becoming higher than the current maximum limit in Australia.

Adult and egg of *Rhyzopertha dominica,* an insect resistant to phosphine, was used as a model insect to study the impact of sorption of SF into the commodity on its efficacy. Bread wheat was exposed to 0.5, 1, and 2 mg/L SF fumigated for 168 h at 25°C and 60% rh. Results indicated a dramatic initial and then gradual chemical sorption of SF into the wheat grain and this sorption affected the toxicity of SF against both egg and adult life stages. However, complete sorption was predicted after 34 d. The major effect of sorption on the mortality of egg and adult related to physical sorption. There was a quadratic relationship between the mortality rate constants of adult and egg and physical sorption and a linear relationship between the mortality rate constants of adult and egg and chemical sorption of SF in wheat. It is suggested that traditionally used CTPs (concentration x time products) need revision and consideration should be given to the sorption of SF by wheat.

Selected samples taken from the fumigation vs. temperature, moisture and SF dosage were evaluated for the effect of SF on the technological characteristics of wheat grains and their derived products (semolina, pasta from durum wheat and bread from hard wheat). SF greatly reduced germination percentage and this effect was enhanced at higher doses of SF and at higher grain moisture content and temperature. The lowest germination was 1.5% at 31.25

mg/L SF, 15% moisture and 35°C compared to unfumigated wheat (90.25%). This is due to the toxicity of SF. Fluoride residues in cereal grain were higher than the maximum residue limit. Milling reduced fluoride residue below the maximum residue limit probably because the majority of fluoride residue is concentrated in bran and this is removed during milling. Cooking pasta also helped reduce fluoride and this moved into the cooking water. Fumigation with SF increased the yellowness of semolina, cooking loss, over cooking tolerance, firmness and stickiness. These factors were affected by temperature with a significant interaction in many cases. However, fumigation with SF at different conditions had no affect on bread making quality. SF affects germination significantly so that it not suitable for grain stored as seeds, in addition to its effect on some quality traits for durum, pasta, and bread. SF may leave fluoride residues in cereal grains higher than the maximum residue limits in Australia if the fumigated commodity was stored with high levels of filling ratios and the fumigation was repeated more than three times at high storage conditions of temperature and moisture. In addition, whole grain or milled product as milled products such as flour and semolina sorb the fumigant more than the whole grain. However, after milling, flour residues levels will be less than the maximum reside limits and the majority of the residues will be in bran.

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PUBLICATIONS RELATED TO THIS THESIS

Mudhir Hwaidi, Patrick J. Collins, Mike Sissons, Hervoika Pavic, Manoj K. Nayak., 2015. Sorption and desorption of sulfuryl fluoride by wheat, flour and semolina. Journal of Stored Products Research 62, 65-73.

ABBRIVIATIONS

a-	Greenness
a*	Red-green difference for colour
a+	Redness
AACC	American Association of Cereal Chemistry
AD	Anno Domini
AEGIC	Australian Export Grain Innovation Centre
AGI	Australian Grain Industry
APVMA	Australian Pesticide and Veterinary Medicines Authority
ATP	Adenosine Triphosphate
b-	Blueness
b*	Yellowness index
b+	Yellowness
°C	Temperature degree
CIMMYT	International Maize and Wheat Improvement Centre
CL	Cooking Loss
cm	Centimetre
CP	Cooked Pasta
CTP	Concentration x Time Products for fumigation calculations
DAF	Department of Agriculture and Fisheries
DNA	Deoxyribonucleic acid
DP	Uncooked Pasta
DPI	Department of Primary Industries
EPA	Environmental Protection Agency
EPPO	European and Mediterranean Plant Protection Organisation

F	Fluoride residue
FAD	Food and Drug Administration
FAO	Food and Agriculture Organisation
FAOSTA	Food and Agriculture Organisation Statistics
FFDAC	Federal Food, Drug and Cosmetic of America
FPD	Flame Photometric Detector
g	Gram for weight
g/ml	grams per millilitre for bulk and grain density
GC	Gas Chromatograph
GIEWS	Global Information and Early Warming System
h	hour
ha	hectare
HLW	Hectolitre Weight
IAEO	International Atomic Energy Agency
ISTA	International Rules of Seed Testing
Kg/hL	Kilogram per hectolitre
Kgy	Kilogray
Kpa	Kilopascal
L*	Brightness for colour
m.c.	Moisture Content
mg.h/L	Milligrams per hour per litre (a unit for CTP)
mg/kg/day	Milligrams per human body weigh per day (allowed for F)
mg/L	Milligrams per Litre (fumigant concentration per volume)
min	Minutes
ml	Millilitre

mm	Millimetre
MPH	Maximum Peak Height
MPT	Mixograph peak Time
mRNAs	Messengers of Ribonucleic Acid
mt	metric tone
NSW	New South Wales
OC	Optimum Cooking Time
OCT	Over Cooking Tolerances
PDS	Public Distribution System
QLD	Queensland
r.h	Relative humidity
RACI	The Royal Australian Chemical Institute Incorporated
RBD	Resistance Break Down
RO	Reveres Osmoses (for water)
Rpm	Revolution per minute
SA	South Australia
Sec	Second
SF	Sulfury Fluoride
SKHI	Single Kernel Harness Index
TAI	Tamworth Agricultural Institute
TCD	Thermal Conductivity Detector
USDA	Unites States Department of Agriculture
V	Volt
VIC	Victoria
vs.	Verses

WA	West Australia
WA8	Width of Mixogram at 8 minutes past peak mixing
WAP	Width of mixogram at Peak time
WHO	World Health Organisation
WI	Whiteness Index for colour
YI	Yellowness Index for colour